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The Analyst,

INCLUDING THE PROCEEDINGS OF

THE "SOCIETY OF PUBLIC ANALYSTS."

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT OF THE
ANALYSIS OF FOOD AND DRUGS, AND OF GENERAL
ANALYTICAL AND MICROSCOPICAL RESEARCH.

EDITED BY

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for Lambeth, Wandsworth, Southwark, Rotherhithe, Newington,
Tenterden, etc., Analyst to the Metropolitan
Asylums Board.

VOL. XIV.

1889.

LONDON:
PUBLISHED FOR THE PROPRIETOR, BY MESSRS. BAILLIERE, TINDALL & COX,
KING WILLIAM STREET, STRAND, W.C

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Printed by HAZELL, WATSON, & VINEY, LD., London and Aylesbury.

THE ANALYST.

JANUARY, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

N ordinary meeting of the Society was held at Burlington House, on Wednesday, the 2th December, the President, Mr. Allen, in the chair.

The minutes of the November meeting were read and confirmed.

Mr. Turner was appointed to act with Mr. Fox in auditing the accounts for the st year.

The following gentlemen were proposed for election:—As members, Mr. T. S. ladding, New York; Professor V. Stein, Government Laboratory, Copenhagen. As sociate, Mr. Sydney Steel, assistant to Mr. Bernard Dyer.

On the ballot papers being opened, the following gentlemen were declared duly ected:—As member, Mr. R. W. Woosnam, London, analyst to the Dairy Supply Comany. As associate, Mr. A. J. Starey, assistant to Mr. L. Briant.

The following papers were read and discussed:—

"On the Determination of Citric Acid in Lemon-juice." By Rowland Williams.

"On some Abnormal Samples of Butter." By the President.

A long discussion ensued, and ultimately a Committee was appointed, as reported on ge 17.

"The Werner-Schmidt Method of Determining Fat in Milk and Cream." By A. W. Stokes.

"On the Examination of Lard for Adulteration." By T. S. Gladding.

"On the Action of Iron Pipes on Water Conveyed Therein." By S. Harvey.

The annual meeting of the Society will be held on the 9th inst., at Burlington ouse, and afterwards the annual dinner will take place. Full particulars will be uly announced.

DETERMINATION OF THE MELTING POINTS OF SOAP-THICKENED OILS.

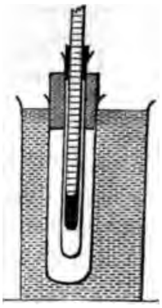
By W. F. K. Stock, F.C.S., F.I.C.

(*Read at Meeting, November*, 1888.)

During the last six months the writer has been engaged in the production of soap-thickened oils. Correct determinations of melting points was a matter of the first importance, but the presence of soap rendered the ordinary methods of working impracticable. The melting points of many samples range far beyond the temperature of boiling water, and the effect of a heat much below the actual melting point is to cause the liberation of a small portion of the oil and entirely vitiate the results of either adhesion or gravitation processes.

Constant readings are obtained by operating as follows:—A clean narrow test-tube is fitted to a thermometer by means of a short length of hard rubber tubing. The space between the walls of the test-tube and the thermometer must not exceed the sixteenth of an inch. The test-tube is passed through a hole in a cork, and this fits the mouth of a wider and longer tube. A little of the sample to be tested is taken upon the bulb of the thermometer, which is then thrust carefully into the narrow tube to within half an inch of the bottom.

The whole arrangement is now suspended so that the wider test-tube is sunk to nearly its entire length in a beaker of clear lard oil. The beaker stands upon an iron plate over a Fletcher Argand lamp—a little manipulation of the gas supply ensures a gradual and regular rise of temperature. The tubes are brought up to the side of the beaker, so that the observation may be taken by aid of a hand lens. The true melting point is accepted as that at which the mass flows down to a clear bead at the bottom of the tube. The writer has obtained exactly concordant results with oils containing 20 per cent. of soap. The accompanying sketch will serve to make the details clear.



THE PRESERVATION OF MILK SAMPLES.

By H. Droop Richmond.

(*Read at the Meeting, November*, 1888.)

The preservation of Milk Samples is most important to public analysts; a milk analysis must be commenced immediately on receipt of the sample, and after the analysis is finished it is not possible to check the results if required, nor should the analysis be disputed, can the sample be referred to another analyst.

Allen (Analyst, XI., 203) has devised a process for the preservation of milk samples by means of alcohol, but as a large quantity of the preservative must be added, very much depends on the exactness of the proportions, and it is doubtful whether the analysis of a sample so preserved would be of any value in a law court.

It is, of course, apparent that as small a quantity as possible of the preservative should be added. Besides carbon bisulphide, and ether, which were used by Hehner, I have tried the preservative action of dichlorophenol, chloroform, terpenes, and hydrofluoric acid; none of these except hydrofluoric acid proved wholly satisfactory; chloroform, which kept the milk in a fluid state, and the fat in an easily miscible condition, being perhaps the most unreliable; carbon disulphide kept the milks fluid, but allowed the cream to rise to form a cake at the top, which could not easily be mixed again; dichlorophenol preserved the milks fairly well, but the cream rose; ether and terpenes were of very little use; hydrofluoric acid, of which ·5 per cent. was added, or one drop to 10 c.c. of milk, preserved the milks almost perfectly; of course it precipitated the casein, but a vigorous shake brought this into so finely a divided state, that sampling was quite easy, the fat being evenly mixed; there was not the slightest blackening of the total solids after preservation, showing that decomposition had not taken place; the fat was estimated by Adams' method (Thomson's modification), there being no difficulty in pipetting 5 c.c. on to a strip of paper, which absorbed it fairly well; in some cases it was necessary to add, after putting the milk on the paper, a few drops of ammonia, and to mix the milk and the ammonia with the stem of the pipette, which was afterwards wiped on a dry portion of the paper; the ash was taken in many cases before and after, and was found to have increased about ·1 per cent.

A table of analyses is subjoined.

No.	Days kept.	T. S. Orig.	T.S. Presvd.	Diff.	Fat Orig.	Fat Presvd.	Diff.
1.	183	8·53	8·57	+·04	2·27	2·21	-·06
	347	„	8·57	+·05	—	—	—
2.	49	12·60	12·60	—	4·16	4·07	-·09
	145	„	12·32	-·28	—	—	—
3.	73	12·56	12·50	-·06	3·58	3·53	-·05
4.	45	13·36	13·17	-·19	—	—	—
5.	38	11·00	11·09	+·09	—	—	—
6.	39	11·56	11·50	-·06	3·23	3·20	-·03
7.	34	13·19	13·15	-·04	5·24	5·27	+·03
8.	34	13·24	13·35	+·11	—	—	—
9.	34	11·45	11·40	-·05	—	—	—
10.	32	11·46	11·50	+·04	—	—	—
11.	32	12·03	12·04	+·01	4·03	3·96	-·07
12.	32	9·77	9·84	+·07	—	—	—
Av.		11·56	11·54	-·02	3·75	3·71	-·04

The whole of these milks were quite fresh when preserved; the following on the contrary were already three days old, when the hydrofluoric acid was added, and many had begun to turn—

No.	Days kept.	T. S Orig.	T. S. Presvd.	Diff.
13.	30	12·97	12·82	-·15
14.	31	12·65	12·77	+·12
15.	31	14·56	13·84	-·72
16.	31	12·92	12·82	-·10
17.	31	13·33	13·27	-·06
18.	31	12·52	12·04	-·48
19.	32	12·30	12·55	+·25
20.	32	12·84	12·23	-·51
21.	32	12·51	12·65	+·14
22.	32	12·72	12·58	-·14

These last samples were evidently decomposed, they smelt bad in many cases, were curdled in a much more lumpy state than the others, and in several fungus could be perceived.

In the first series, Nos. 1 and 2 after being analysed when preserved, were sealed up again, and kept for a further period; in both cases there was a blackening of the total solids, indicating that some decomposition had set in; this, and the facts that the ash increases, and that the total solids are not blackened when freshly preserved with hydrofluoric acid shows that the hydrofluoric acid disappears as such.

To further test this question a sample of milk was preserved when fresh; another portion was allowed to stand for three days, and then preserved; both samples were again analysed after the expiration of a fortnight, with the following results.

		T. S. Orig.	T. S. Preserved.	Diff.
23.	Milk fresh	11·32	11·40	+·08
24.	After standing 3 days, without hydrofluoric acid	11·07	10·70	−·37

This shows that hydrofluoric acid only exercises a preserving action when added while the samples are fresh; if decomposition has commenced it is of very little use.

I would recommend that a portion of all milks it is desired to preserve, be taken on receipt of the sample, ·5 per cent. of hydrofluoric acid be added, and the bottle securely sealed; no correction need be made for dilution of so small a quantity of liquid, as the difference is well within the experimental error.

The antiseptic power of fluorine compounds was pointed out by W. Thomson in a paper read before the British Association last year.

In conclusion, I must express my obligation to Mr. Hehner, in whose laboratory these experiments were carried out, for his advice and kindly interest in the work.

The Laboratory, 11, Billiter Square, E.C.
November 14th, 1888.

Discussion.

Mr. **Davies** said he thought the suggestion was decidedly valuable. He would be glad to know whether by the statement that one-half per cent. hydrofluoric acid should be added to the sample it was intended to add the ordinary acid of commerce in this proportion, or such an amount of this solution as would give one-half per cent. true acid in the resulting mixture.

Mr. **Blount** asked if the acid would have any corroding effect on the glass vessels employed?

Dr. **Vieth** said he was rather adverse to any tampering with samples, and it would be tampering if one-half per cent. of hydrofluoric acid or anything else were added. They would not like an inspector to add the acid, and if added, when the sample came into the hands of the public analyst it would have nothing to do with the sample kept for reference. He did not think a public analyst would recommend the addition of even a small proportion of acids.

Mr. **Hehner** said that some time ago he brought some experiments before the Society and the same point was raised as to the desirability of preserving milk samples. No doubt it was very undesirable to give inspectors power to add anything to samples, and Mr. Richmond was adverse to that; but in the laboratory, and especially during the summer, it was much better in emergencies to be able to preserve samples without fear of their going bad than not to be able to do anything at all. He certainly thought that every sample which was condemned by the public analyst should be kept by him for reference. Not that portion which might go to Somerset House, as they had nothing to do with that, but their own sample should be preserved. He had seen all these experiments of Mr. Richmond's—the milk was curdled by the hydrofluoric acid—

but a good shake produced a perfect emulsion. There was no difficulty whatever in analysing it. In the experiments which he brought before them a year ago, in no case was the milk so old as those of Mr. Richmond's.

Mr. Richmond in reply, said the hydrofluoric acid he used was the ordinary acid of commerce, and that he added ·5 per cent. of that and not of the actual hydrogen fluoride. As regards Mr. Blount's question referring to the corroding effect of the acid on the vessels employed, he was not quite certain—if he meant had it any corroding effect on the glass bottles in which the milk was kept—he believed it had; but if he meant the porcelain basins used in the analysis, it had not. The weight of the basins after being cleaned out was exactly the same as before. As regards Dr. Vieth's objections, they had been answered by Mr. Hehner, with whose remarks he agreed.

[Note.—We have received the following from Dr. Vieth:—In the remarks which I made in the discussion on Mr. H. D. Richmond's paper "On the Preservation of Milk Samples," I have hardly done justice to this paper. Having fixed my mind upon one particular point, I must confess that in the discussion I lost sight of others. I am very strongly and most decidedly of opinion that official samples kept for reference should not deliberately be materially altered in their composition, not even for the sake of increasing their keeping qualities. But that does not exclude in other instances the employment of a convenient method for preserving milk samples in a state which allows their analysis to be deferred to a future time without interfering with the correctness of the results. Looking at Mr. Richmond's experiments, I freely admit that his suggestion, *i.e.*, preserving milk samples by the addition of a small definite quantity of hydrofluoric acid, is very valuable, and seeing that he has obtained exceedingly well-agreeing results in samples when analysed fresh and after having been kept for weeks, and even months, certainly one which is worth trying by analysts who in their private practice may receive milk samples with which they cannot deal at once.]

ON SOME ABNORMAL SAMPLES OF BUTTER.

By Alfred H. Allen.

(*Read at Meeting, December,* 1888.)

In July last a sample of butter was condemned by a Public Analyst as being adulterated. The case was referred to Somerset House, and the chemists there, after enquiring as to the terms of the report of the Public Analyst, confirmed his certificate. The butter in question was shipped to England from Copenhagen, but was not of Danish origin, the dairy being that of Kjellstorp, near Wernamo, in Sweden. On learning that the butter had been condemned, the shipper sent a sample of butter from the same dairy to Professor V. Stein, the official analyst to the Danish Government. At the same time he took measures to have the dairy visited by the King's Bailiff and the British Vice-Consul, who went there and examined everything in and about the dairy, and also interviewed the owner and persons employed in the dairy, all of whom swore that no adulteration of the butter had ever taken place. In due course Professor Stein reported that the sample sent him contained so small an amount of volatile acids as to justify him in calling into question the genuine nature of the butter. At his request the King's Bailiff again proceeded to the dairy to see the butter made and to take samples of it. He watched the process of churning and working the butter in the presence of a representative of the British Vice-Consul, and, as the result of the subsequent analysis, this sample of butter was found to have characters practically similar to those previously observed in the butter sampled in Copenhagen and in the butter condemned in England, some of which had been sent to Professor Stein. On learning the history of the sample of butter from the dairy, Professor Stein was led to doubt how far the limits generally adopted by public analysts in the examination of butter were to be relied on.

The Royal Agricultural Society of Denmark, which, in many respects, acts as a branch of the Danish Home Office, now became fully alive to the importance of the question at issue, for Denmark being practically an enormous dairy-farm—a large proportion of the produce of which is sent to England—it was felt that the abnormality which had been observed in the case of the Swedish butter might very possibly extend to certain Danish butters, and hence it was very important to know the variations in composition which were natural to butter under exceptional circumstances. In support of this view, and at the suggestion of Mr. Harald Faber, Agricultural Commissioner abroad to the Danish Government, I was formally invited to visit Denmark, inspect thoroughly the whole process of the manufacture of butter at certain dairy-farms, and analyse the product obtained under these crucial conditions. Of course, it would have been impossible to visit and watch the process of butter-making at even a tithe of the numerous dairy-farms existing in Denmark, but the investigation was simplified by the fact that Professor Stein, who practically occupies the position of Public Analyst for the whole of Denmark, receives, under the Danish Margarine Act, three samples of butter daily, three inspectors being constantly employed in visiting the farms and taking samples of the products. Hence, Professor Stein was able at once to point to certain farms whence very abnormal butter had been obtained, and consequently our attention was specially directed to the product from these places. Accordingly, early in last month I visited Copenhagen, and met in conference at the Government laboratory Professor V. Stein; Professor Th. R. Segelcke, Lecturer on Dairy-products in the Copenhagen College of Agriculture; Mr. B. Böggild, Consulting Dairy Chemist to the Royal Agricultural Society of Denmark; and my travelling companion, Mr. Harald Faber, Commissioner Abroad of the Agricultural Department of the Danish Government. The following day we (with the exception of Professor Segelcke, and with the addition of Mr. A. Stewart MacGregor, British Vice-Consul at Copenhagen) proceeded to a distant part of Denmark, where was situated one of the farms ("B") already referred to. There we witnessed the milking of thirty-one cows, the product from each of which was weighed separately. The mixed milk was then put into a receptacle for ripening, and the lid securely sealed and fastened by Mr. MacGregor and myself. The next morning, having noted the conditions of the seals, we opened the vessel, and watched the transferance of the contents to the churn. The usual addition of butter-colouring was omitted. The churning being completed, we watched the butter worked and placed in a number of tin boxes, which boxes were then put into strong linen envelopes, which were closed and sealed by Mr. MacGregor and myself. We then proceeded to another farm ("O"), where we saw sixty-five cows milked, and watched the manufacture of butter in the same jealous manner as before, the product being duly sealed as on the previous occasion. The milking of the cows, as well as the treatment of the milk and butter, was conducted at both farms under the unremitting supervision of the whole party, and especially of Mr. Macgregor and myself. I am, therefore, in a position to give a most positive guarantee that the samples of butter in question were absolutely genuine products.

The butter made at "B" farm was the produce from milk yielded by cows which had been in the stalls for six days, but which previously had been fed on grass land reclaimed from the sea. Most, but not all, of the cows were in calf, some being pretty far advanced, but others not expected to calve until May next. At "O" farm the cows had been longer in the stalls, but were all expected to calve within a few months.

I may say here that I was greatly struck with the extremely methodical manner in which the operations of milking and butter-making, and in fact the whole work of the farms, were carried out. Everything was scrupulously clean, and the whole manufacture was conducted on the most scientific principles, a thermometer being employed at every stage of the operation, the milk and other products weighed, and a definite proportion of salt added. It is impossible to conceive a manufacturing operation of the kind carried out in a more perfect, systematic, and cleanly manner, and it certainly is a matter of regret that many of our English dairies are so far behind those in Denmark.

On returning to Copenhagen, Professor Stein and I opened one of each of the sealed packets and jointly made an analysis of the contents. The method on which we placed the most reliance was Wollny's modification of the Reichert Process for determining the volatile acids," a translation of which by Mr. O. Hehner was published in the ANALYST last winter. The process as there described (Vol. xiii. page 40) is strictly that used by Professor Stein, but I may say that he lays special stress on the importance of ensuring the entire absence of carbonic acid. The water used is hot or recently boiled, and, by means of a T-piece in the tube by which the flask is connected with the condenser, the alcohol can be distilled off, and water added to the residual soap without opening the flask and so exposing the contents to the air. Another important point to which great attention is directed is to heat the acidulated liquid very gradually, and not to attempt to commence the distillation until the layer of fatty acids has become clear and the aqueous liquid has almost entirely lost its original milky appearance. The time of thirty minutes for the distillation of 110 c.c. is strictly adhered to, with a variation of not more than two minutes more or less.

My previous experience of Wollny's method, which I have used habitually for the analysis of butters ever since the appearance of the translation in ANALYST a year ago, had shown me that the results were very satisfactory, and, if anything, somewhat higher than those I used to obtain by the Reichert process as worked by myself, and described in my "Commercial Organic Analysis" (Vol. II. p. 45). The higher result is probably due to two or three causes. Thus, I had been in the habit of distilling off 50 c.c. out of 75, which is a smaller proportion than 110 out of 140. This difference probably more than compensated for any error caused by the presence of carbonic acid in the old way of operating. As worked by me, I do not think I ever lost volatile acids in the form of butyric ether, as I always used a flask fitted with a proper condensing arrangement. Professor Stein's experience of a number of samples of butter of known purity shows that, in his hands, and worked in the exact manner I have described, the volume of decinormal alkali neutralised by the volatile acids from 5 grammes of butter-fat ranges from 31·59 c.c. to 25·08 c.c. with an average of 28·64. I should myself have fixed the average at 28·00, or a little below it, so that our experience is fairly concordant in that respect.

The experiments on the samples of butter-fat in question, which were marked respectively " B " and " O," gave in Professor Stein's laboratory the following results:— " B " required 22·70 c.c. which, with the correction of 0·17 shown by a blank experiment, gives 22·53 c.c. as the final result. The butter-fat from the sample marked " O " gave a result of 24·76 c.c.

I may say that I took the most rigid precautions to avoid the introduction of any possible source of error—such precautions as might well have been resented by a brother chemist, had he not been well aware that their object was to meet all possible criticisms, and not because there was any real doubt in my mind respecting the conditions of working. Thus, I proved the accuracy of the weights of the balance employed; I personally took every weighing and measurement; I proved the neutrality of the distilled water, alcohol, and phenol-phthalein employed; I verified the capacity of the burette, pipettes, and measuring flasks; and I set the baryta solution. This was done by titration, with a sample of quadroxalate of potassium obtained from Professor Wollny, of Kiel. I brought some of this reagent home with me, and have since verified its substantial, though not absolute, accuracy, by titration against solutions made up in England. I have also made for myself some of this acid oxalate of potassium which has the formula KHC_2O_4, $H_2C_2O_4 + 2$ aqua, and seems to offer certain practical advantages over oxalic acid, as it is of definite composition, easily prepared and purified, and not efflorescent. I have prepared it by making a saturated solution of re-crystallised oxalic acid in water and filtering the liquid. One-fourth of this liquid was then heated to boiling, and neutralised with pure potassium carbonate, employing litmus paper as an indicator of neutrality. This neutralised solution was then added to the main quantity, whereupon a copious deposition took place of the quadroxalate in the form of a granular powder. This powder is filtered off, washed with a little cold water, and dried over sulphuric acid. In using the quadroxalate for setting the baryta solution, about ·25 of a grm. is exactly weighed and dissolved in a little warm water. Phenol-phthalein is then added and the baryta water run in until the pink colour becomes permanent. During the first part of the titration the solution remains quite clear, but subsequently a white precipitate of oxalate of barium is produced, and this forms an extremely delicate background for viewing the end of the reaction. In fact, I think it would be a distinct advantage in such titrations to aim at the presence of a finely-divided white precipitate in the liquid, rather than to employ a white background to the vessel. Of course, baryta possesses a manifest advantage for this purpose over caustic soda, for a clear solution of baryta must be free from carbonic acid, the presence of which affects any titrations in which phenol-phthalein is employed as an indication.

The time at my disposal in Copenhagen did not allow of any further analytical examination on the spot of the butter-fats in question, with the exception of the determination of the specific gravity of the sample marked " B." This was ascertained by a specific gravity bottle of a kind I have been unable to obtain in England. It was a conical bottle, furnished with a thermometer having a range of a few degrees above and below 100° C. There was also a second orifice, consisting of a vertical capillary tube ending at the top in a small funnel. The bottle having been filled with the butter-fat and the thermometer stopper inserted, it was immersed in a beaker of water, the temperature of which did not rise more rapidly than 1° C. per minute, so that there was ample time for the contents of the bottle to acquire the temperature of the surrounding water. When an external thermometer shewed this to be at 100° F., the fat which had passed by expansion into the little funnel surmounting the capillary tube was removed by means of filter paper, so that the measure of the fat was accurately adjusted to a mark just at the top of the capillary tube. The bottle was then removed and weighed with its contents, after which it was returned to the water and the heating continued, until the internal thermometer indicated that the fat had reached a temperature of 100° C. It was then removed and weighed as before. We had previously observed the weight of the bottle when dry and empty and when filled with water at 15° C. Hence the experiment gave us the means of ascertaining the density of the butter not only by Bell's method at 100° F., but also at 100° C.

The following were the figures obtained, water at 15° C. being unity:—

Specific gravity of "B" butter-fat at 100° F. (=37·8° C.) ·9055.
" " " 100° C. (=212° F.) ·8639.

This gives an apparent expansion-coefficient of 0·00067 for 1° C.

In concluding the description of the experiments made at Copenhagen, I cannot omit to give expression to my sense of the great courtesy shown me by Professor Stein and his assistants, and my high appreciation of the methodical manner in which the work there is evidently done, and which is in itself so good a guarantee of its accuracy.

It must be remembered that in order to ripen the milk in time to allow of its being churned in twelve or sixteen hours after milking, it is necessary to add to it a certain proportion of buttermilk. Hence my critical examination of the butters in question would not have been complete had we omitted to analyse the butter-milk added, with a view of proving that it was not a medium of introduction of any foreign fat. The examination was made by rendering 100 c.c. of the butter-milk alkaline with ammonia and agitating with ether. The ether was allowed to separate and the agitation repeated with a fresh quantity of ether. The ethereal solutions were mixed and evaporated, the residual butter-fat being weighed. It amounted to barely 0·1 per cent. of the butter-milk, and hence even had the fat so extracted consisted wholly of foreign fat, it could not have appreciably affected the results of the analysis. As a matter of fact, however, we made an observation of the index of refraction of the extracted fat by means of Abbé's Refractometer, and found it to be 1·4620—a result which agrees with that of genuine butter-fat. I may here say that this is my first experience of the use of Abbé's Refractometer, which Professor Stein informs me is capable of distinguishing with certainty butter from other fats, but is not of much service for mixtures containing butter.

I brought away with me some of the sealed packets containing samples of the butter in question, while others were left with Professor Stein and Mr. Faber. I also brought a portion of the butter-fat rendered from the butters in question in Professor Stein's laboratory. Since my return to England I have analysed these samples of butter-fat as well as the butters brought home in their original condition, and have not limited myself merely to the Reichert-Wollny process, but have examined them by other well-known processes.

My results are as follows :—

	B.		O.		N.
	Butter-fat Rendered in		Butter-fat Rendered in		Average butter from V. Stei
	Copenhagen.	London.	Copenhagen.	London.	
Specific Gravity at 100° C.	·8639	·8640	·8641	·8641	·8653
Decinormal Alkali by Reichert - Wollny Process	22·63	22·39	24·39	24·70	29·2
Percentage of KHO required	22·27	22 05	22·15	22·03	22·67
= Saponification Equivalent ..	251·9	254·4	253·3	254·6	247·4
Soluble Fatty Acids, per cent... ..		4·37 4·50 4·45		4·60 4·66 4·77	5·52 5·54
Reichert-Wollny on Soluble Acids ..		22·76		24·70	28·96
Insoluble Fatty Acids per cent... ..		90·24 90·62		89·90 90·30	88·47 88·49
Melting Point of Insoluble Acids ° C		36·5		39·0	41·0
Turbidity - temperature ; Acetic Acid Test : — Genuine Butters 62-63° ..	74·5	75·0	72·5	73·0	68·5

Before proceeding to the discussion of the other results, some explanation is requ respecting the above determinations of specific gravity. The figures quoted are ca lated to a temperature of 100° C., though actually observed at a temperature of 99·5 The coefficient of expansion of butter-fat according to the experiment already reco being ·007 for 1° C., it follows that the gravity of, say "N" sample at 99·5° was ·86 and similarly the other observations were ·00035 higher than the figures recorded in tables.

In my work on "Commercial Organic Analysis," Vol. II., I state the ave specific gravity of butter-fat as being 0·868 at 99°, equivalent to ·8673 at 100°. determination, like nearly all the other densities of fats at 100° C. published by was obtained by the means of a Westphal balance. The accuracy of this had certained by comparing the gravities of proof-spirit and milk shown by it with de ion of the gravities of the same samples made by the specific gravity bottle, and cy of the rider weights was also verified—a precaution which was by no m unnecessary, as one of the riders was found to differ very seriously from its supposed weight. Very recently I was struck with certain differences in the determinations of densities of butter, lard, and other fats at the boiling point of water when made by the plummet and by the specific gravity bottle or Sprengel-tube, and for some time was wholly at a loss to account for the discrepancy, which, however, has now been definitely traced to the faulty division of the beam of the Westphal balance. The instrument I used was one purchased of a London firm that has supplied a number of instruments in this country. Mr. W. Chattaway, who discovered the nature of the error, has examined all those in stock at present, and finds the error common to all, and therefore very probably it exists in all or most similar instruments in use in England. The fault consists in the notch at the eighth division of the beam being sensibly nearer the fulcrum than it should be. The effect of this is evident. All weights placed on the eighth division do not exert their full leverage, and additional weights have to be added to effect the counterpoise. In the case of my instrument, the error can be readily detected with a fine pair of compasses, and is even visible to the unassisted eye of some observers when their attention is directed to the point. The consequence is that the greater number of the determinations of gravity, commencing with ·8, including all determinations of specific gravities of butter, lard, and other fats at 100° made and published by me up to a very recent date, have been in excess of the truth by about ·0015. Hence the statement in my book that the average density of butter at 99° is 0·868, should be modified to 0·8665 = 0·8658 at 100°. The discovery of the error in question would have been made long ago had the balance been checked against the specific gravity-bottle with rectified spirit instead of proof spirit and milk. The history of it I have given will serve as an illustration of the ease with which an error may creep into absolute determinations, and hence how difficult it is for chemists to compare strictly their own results with those obtained in other laboratories. Of course all my results are strictly comparable among themselves, so that the error has not affected any conclusion arrived at; but I think it right to place the fact on record, as I believe other chemists are likely to be obtaining erroneous figures from exactly the same cause. One other practical conclusion may be deduced from my experience, and that is, that the rider system is open to grave objections when applied to the larger weights of a balance.

With regard to the determination of the saponification equivalent and of the soluble and insoluble fatty acids, I have nothing to say, except that the highest estimations of insoluble acids were obtained in experiments in which the washing was known to be carried out less perfectly than in the other cases.

The turbidity-temperatures by the acetic acid test were made by heating 3 c.c. of the melted butter-fat with its own measure of glacial acetic acid until the mixture was clear, and then stirring with a thermometer until the liquid became turbid throughout. The test-tube in which the experiment is conducted should be immersed in an empty flask, so as to avoid too sudden cooling and the effects of currents of air. I have now used this test for several years with very satisfactory results. It enables butter to be distinguished from other fats with the utmost facility, and in the case of samples of genuine butter, the temperature at which turbidity occurs closely follows the other characters of the butter; that is to say, a butter rich in lower fatty acids, and therefore giving a high result by the Reichert-Wollny test, will give a low turbidity-temperature by the acetic acid test. Repeated experiments on the same samples give results which are concordant within 1° C. The strength of the acetic acid used affects the test in an enormous degree; in fact, I fancy the solubility of a definite fat in glacial acetic acid would be the best possible test for the strength of such acid. A sample of acid which when used for testing genuine butter will show a turbidity-temperature of 60° C. will show with butterine a turbidity-temperature of 100°, but by putting a very small proportion of water to such acid the temperature of turbidity is so raised that the results of the butter-test will be like those given in the table. Hence, in order that the results may be comparable, it is necessary to define in the strictest manner the strength of acid used, and this is somewhat difficult to do. The acid I employed for the experiments quoted above had a specific gravity of 1·0563 at 15·5° C., and when titrated with standard baryta water and phenol-phthalein, was found to contain 95·14 per cent. of real acetic acid ($C_2H_4O_2$).

From a careful consideration of the foregoing experiments, it appears that the butter in question marked "B" had a density quite outside previous experience of genuine butter-fat, and by analysts relying much or exclusively on this indication would be very liable to be condemned as adulterated. The volume of alkali required by the butter when examined by the Reichert-Wollny process was sensibly below the limit hitherto recognised. The saponification-equivalent was at or beyond the extreme limit hitherto observed (253). The proportion of soluble fatty acids was lower than has been observed in any previous genuine butter, except some analyses of doubtful accuracy. The insoluble fatty acids are sensibly in excess of those previously observed; the highest figure recorded by Dr. James Bell in a butter not altered by keeping being 89·9 per cent. The foregoing remarks apply, though somewhat less strongly, to the sample marked "O." Before leaving this part of the subject, I may say that I have on the table a further quantity of the butters in question contained in the original sealed packets. I have permission to place them at the disposal of any Public Analyst who will undertake to examine them fully and communicate his results to me.

Although the Danish butters which yielded me the abnormal results already described are of exceptional character, we must remember that they really represent the butter produced from the mixed milk of a large number of cows, which were yielding much the same kind of butter a year previously, and in the face of these facts, and of the Swedish experience already recorded, it cannot be said that such butter may not be met with in practice.

My attention has been called by Mr. Faber to some experiments made by Adolph Meyer, published in the "Milch-Zeitung" for October 17. These relate to the butter produced by a particular cow which, according to Meyer, yielded milk the butter-fat of which had the following characters.—

	Specific gravity at 100°.	Volatile acids c.c.
1st Hay period	·8632	29·5
2nd " "	·8627	27·1
1st Ensilage period	·8621	26·4
2nd " "	·8620	20·2
1st Mangold "	·8640	32·4
2nd " "	·8629	28·2

Meyer states, however, in a foot-note, that the specific gravity figures are merely comparative, not absolute; so that the results have less value than would otherwise have been the case.

Nilson, of Stockholm, again, in experiments made in 18°6 by the unmodified Reichert process, found the volatile acids to vary materially with the length of time since calving. While very low at first, requiring only about 10 c.c. (+2 = 20 for 5 grms.) of alkali for their neutralization, they rose to a maximum in about a week, and maintained this high figure for about four weeks, after which they fell gradually to about 12 (= 24), but only very rarely below that figure.

The produce from single cows may, of course, be expected to vary still more than that from a number of animals, but in practice there must be very little butter sold which is the produce of only one or two animals, and the milk of a normal cow being usually much larger in quantity than that of an animal yielding an abnormal product, the variations from the mean composition in the case of united milk is not likely to be great.

A very great object would be gained, if we could trace a sample of butter back to the farm and cows which produced the milk, and I think we should look forward to the day when an alteration of the law will render this possible.

Although the results I have recorded undoubtedly show that butter-fat varies in composition more than was supposed, and will render the detection of adulteration decidedly more difficult in cases where a moderate percentage of foreign fat has been added, I do not think we have yet come to the end of our powers in butter analysis. The detection of cocoanut oil, which I met with in one sample of butter in considerable amount, can be effected with certainty, by the large yield of solid volatile acids and their peculiar odour. Besides, the specific gravity of the butter is rather increased than diminished by the presence of cocoa-nut oil. Cotton-seed oil will indicate its presence in more than one way. Seeing that all margarine now-a-days contains cotton-seed or other vegetable oil, it is worth while to try and detect their presence. The isolation of phytostearin, or vegetable cholesterin, might possibly afford a test for vegetable oils, as suggested by Mr. Hehner with respect to lard. Some modification of the chloride of sulphur test may be found available, while cotton-seed and most vegetable oils might be indicated by the increased iodine-absorption of the sample or of the oil separated from it by pressure. In such expressed oil, earthnut oil might possibly be recognised by isolating arachidic acid, while sesame oil can be detected by the sugar reaction even in the original sample. The chemistry of the vegetable oils is still imperfectly understood, and researches in this direction would almost certainly lead to the discovery of new methods of detecting them. Hence, we ought to aim at a further advance in our methods of examining butter, rather than remain content with processes which, however good and ingenious in their way, do not give us all the information we require.

Professor Wollny, of Kiel, has requested nearly thirty chemists to assist him in ascertaining the natural variations in analytical characters of butter-fat, in order to render the results more strictly comparable. He has published and circulated a printed description of the exact method to be employed in the analysis, together with forms on which the results are to be tabulated. To give an idea of the field proposed to be covered by the scheme, I may quote the following data with which Dr. Wollny requests each analyst to furnish him:—The strength of the sulphuric acid with which the baryta solution is set. The number of c.c. of baryta used for neutralisation. The strength deduced from this result, and the strength of the baryta solution deduced from its titration by acid oxalate of potassium. In estimating the volatile acids, the result of a saponification with and without alcohol. The time of each distillation, and blank analyses in each case. The mean molecular weight of the volatile fatty acids (distilled from their magnesium and copper salts). The volume of baryta solution neutralised by the acids soluble in boiling water and in 10 per cent. alcohol. The percentage of insoluble acids, by washing on the filter and by washing in a current of steam; and their mean molecular weight. The percentage of free fatty acids in the butter-fat. The saponification-equivalent of the fat. The molecular weight of the total fatty acids. The iodine absorption. The refraction index of the butter-fat; and, for comparison, of olive oil, nitrobenzene and alpha-monobrom-naphthalene. The special gravity at 0° or 15° and at the temperature of boiling water, by the specific gravity bottle, plummet method, and hydrometer. These determinations, with the melting point and solidifying point of the fat, seem a tolerably complete examination. It would be impossible, even were it desirable, for our Society to institute any series of experiments of the same wide-reaching character; but it occurs to me that we might with advantage appoint a small committee to receive and tabulate results which might be communicated to them by members of the Society, and to devise, if possible, some method of examining butter which should be less dependent on the natural variations in its composition than the methods at present in use.

In conclusion, I desire to express my indebtedness to Mr. Faber for his good offices as interpreter and travelling-companion, and to Mr. Wm. Chattaway for the zeal and care with which he has executed many of the determinations, the results of which I have recorded.

DISCUSSION.

DR. **VIETH** (who had temporarily taken the chair) said they would all agree with him that the question Mr. Allen had brought forward was of the greatest importance to public analysts. Personally, he was very pleased to see that Mr. Allen had not only taken the trouble to see the butter made, but had seen the cows milked and the milk locked up, so that no hitch could occur similar to that which happened in the case of the Swedish dairy in which the butter was produced, which was the cause of the investigations dealt with in Mr. Allen's paper. In this case the Government's officers only saw the cream churned, and had taken no precaution to put the purity of the cream beyond doubt. Next to milk, butter had given them perhaps the greatest amount of trouble lately.

As regards the requirements of the German Commission which had been mentioned, he should like to add that, not only one, but five determinations of each kind were to be given, and this, of course, caused a great sacrifice of time. Had Mr. Allen made any experiments as far as regarded the distilling off of the volatile fatty acids? Wollny and Stein laid much stress on the fact that distillation should not be proceeded with too quickly, but that the fatty acids should be melted first, and the aqueous liquids allowed to become clear. What difference would it make if they proceeded a little more quickly, and did not wait for the clearance of the aqueous liquid? As far as his experience went he did not think there was much difference. He had found that the butter sent to the members of the German Commission, which was produced at Kiel in July, required about 30 c.c. $\frac{N}{10}$ alkali—Reichert-Wollny—while a number of samples of French and Danish butters, examined during the last weeks, required from 25 to 26 c.c.

Mr. **HARALD FABER** said—It is not to be denied that the proved existence of genuine butter of such an abnormal composition as those mentioned by our president interferes very seriously with the detection of mixtures of butter with foreign fat, and it might therefore seem peculiar at first that an exertion has been made on behalf of the butter producers in Denmark, being very far from interested in mixtures, to put such a check on the control of butter. But I think the position of the Royal Agricultural Society of Denmark to this matter is very clear. A recent case, which Mr. Allen mentioned, proved what risk we run by leaving matters as they were. I wish to point out that we have in no way questioned the correctness of the English analyses of the butter sampled in Wigan and condemned as adulterated; but having had the opportunity of examining samples from the dairy from which the butter in question originated, and some of the butter fat rendered from the official duplicate sample, and having learned already last year that genuine butter can be produced of still more abnormal composition than the butter in question, we expressed the opinion that the fact of this butter having such a composition was not sufficient proof of its being adulterated. But our assertion that genuine butter might be of such a quality that English analysts would call it adulterated was, perhaps, not likely to be taken notice of without further proof. Knowing that some day genuine Danish butter might be condemned here as adulterated if no step was taken, two courses were open to us—either to leave matters as they were, which meant running the risk of losing the well-merited name as producers of first-class and genuine butter, or to prove the existence of the abnormal butter, and then, perhaps, have to compete still more with mixtures from other countries, which would then be more difficult to detect. I urged the adoption of the latter course, which was finally agreed to, and we have all reason to be well satisfied that Mr. Allen, at our invitation, undertook the long journey to Denmark, and also with the excellent work he has done. The way in which these abnormal butters were first found is as follows:—As it has been said, Prof. Stein is the Public Analyst for the whole of Denmark, which is equivalent to about half of London as regards population. He received in 1887 some samples of butter of which he at first felt convinced that they were mixtures. He therefore ascertained the names of the farms from where they originated, and he had samples taken with all guarantee of purity, and in that way found that in September, 1887, some large farms in Denmark, with large herds of milch cows, gave butter which yielded below 25 c.c., by Reichert-Wollny, one even as low as 21·7, another 21·9. His experience in butter analysis is now, as mentioned by Mr. Allen, very extended, having three samples a day from our inspectors under the Margarine Act. Whenever a sample of doubtful character has been taken at a dairy, a warehouse, or a shop, the inspector is sent at once to the dairy where the butter was produced, to obtain an authentic sample. In this way it has been found that butter of this abnormal character may occur at different farms, but, as it seems, for a very limited period only. The lowest figure by the Reichert-Wollny test yet found in butter-fat from a herd is, as far as I know, 21 c.c. It is only this extended experience of Prof. Stein's that enabled him to pick out a farm where we could invite Mr. Allen to go and take samples, and it was only done with great difficulty. Some of the farms where the butter had been abnormal in September, 1887, gave October of this year butter in every respect normal, others gave abnormal butter when first tested, but when the test was repeated a week later, the butter was good again. It was very much of a chance that we hit on a farm yielding butter of a description like the sample "B."

I have been informed by my friend Dr. O. Hehner, that Prof. Besana, in Italy, has proved the existence in Italy of genuine butter of the same abnormal condition. He found amongst 114 samples 2 yielding between 21·8 and 22 c.c. by the Reichert-Wollny process, 1 yielding between 23 and 24, 2 between 24 and 25. It may, therefore, reasonably be expected that such abnormal butter may occasionally be produced even in other countries. It seems happily to be rare exceptions, and this in a double meaning. Not only are such butters met with only on a few farms, but they are found there only at certain times. The reason of this peculiar quality of the butter cannot be given yet; but two circumstances at least seem to have some effect on the percentage of volatile acids. One is, the condition of the field or meadow, another, the distance from the time of calving. Very poor grass land may have the effect, but grass on reclaimed land, although it could by no means be called poor, neither the land nor the grass, and although the grass was plentiful, have in some cases been considered the cause of the low contents of volatile acids in the butter. As far back as 1886, Prof. Nilson, of Stockholm, experimenting on a large number of single cows, found that the contents of volatile acids varied with the distance from the time of calving; on the first days the contents was very low indeed—10 c.c. by the old Reichert test; in about a week it reached a maximum, where it remained for a few weeks, when it gradually fell towards the end of the time of lactation. In this connection it will be of some interest to learn that the cows on farm B which gave the most abnormal butter had, until some days before the samples were taken, been on grass on reclaimed land *of good quality*. They were most of them in calf, but with a [illegible] in time till their next calving of 5 months; while the cows on farm O were all very near to their next calving, and consequently more late milking, but they had not been on reclaimed land.

It ought to be mentioned that low contents of volatile acids seems to stand in no relation whatever to the commercial value of the butter.

Mr. Hehner said that the facts brought forward by Mr. Allen were, without doubt of far-reaching importance. Public analysts would have to acknowledge that exceptional samples of butter did occur which would analyse like mixtures containing about 25 per cent. of margarine. If only one such exceptional sample occurred in every hundred, as there was some evidence from the Italian experiment quoted by Dr. Faber, it would yet be henceforth impossible to give a satisfactory opinion on any sample which contained less than 25 per cent. of foreign fat. Such an acknowledgment would result in immense injury to the public and the butter trade; It therefore behoved public analysts at once to set to work to find out the cause of the extraordinary discrepancy, less in the physiological direction, or regarding the food or other circumstances influencing the cows, but in a chemical direction. The question had to be solved: In what points does a natural abnormal butter differ from one adulterated with, say 25 per cent. of foreign fat? He was convinced that there were such differences, and he drew, in this direction, attention to the following facts, deducible from Mr. Allen's analysis. On calculating from the Reichert results the percentage of volatile acids as butyric acid (although, of course, the totality of volatile acids is not obtained by distillation), there in normal butters almost reached the soluble acids estimated in other ways. Thus, in the "normal" butter of Mr. Allen, butyric acid, by Reichert, 5·14 per cent. soluble fatty acids, 5·54 per cent., or 93 per cent. of the total soluble fatty acid obtained by distillation. In sample O, which would be passed as genuine upon the Reichert results, 4·34 per cent. were obtained by distillation, against 4 66 total soluble acids, or also 93 per cent. But in sample B, which was quite abnormal, only 87 per cent. of the soluble acids were volatile.

These figures seem plainly to indicate that, in this particular abnormal sample at least, the breaking down of the albuminous matter from which butter-fat in all probability results, was not so complete as to form butyric acid, but other acids slightly higher up in the fatty series. This conclusion, he thought, was confirmed by an observation of his (Mr. Hehner's) upon the abnormal sample, a portion of which had been given him by Dr. Faber, namely, that towards the end of the distillation, notable quantities of solid fatty acids congealed in the condenser, similarly as in the case of cocoa-nut oil.

It appeared to him to be of the highest importance now to direct our attention to the intermediate fatty acids, and he had no doubt that thus this difficulty might be overcome. The German Committee would thoroughly investigate the methods at present at our command, and it was not necessary to work in this direction; he would, however, suggest that, unless Mr. Allen was prepared to take personally upon himself the immense labour of carrying out an investigation such as he (Mr. Hehner) had indicated, it would be well that the Society should appoint a Committee upon whom the labour would devolve.

Mr. Allen said in reply that the question was quite a different one from that submitted to the Milk Committee. In the latter case they knew what they wanted, and their object was to devise a method which would give accurate and reliable results in the hands of a number of chemists. In the present case they did not know exactly in what direction to look for success. It was a question of studying more closely the composition of butter as compared with other fats. He had already indicated certain directions in which hopeful work might be done, and he was thoroughly in accord with Mr. Hehner in his suggestion regarding the desirability in future of differentiating the fatty acids more thoroughly than had been done in the past. In fact, he had suggested that direction, as a desirable one in which to work, to Mr. Faber, only a few days ago. His proposal was, that a small committee should be appointed to consider whether they could not devise some other process of examining butter for foreign fats. He did not personally know how far the results were affected by the time of distillation, and by neglect of the precaution to allow the fatty acids to become thoroughly molten before commencing to distil. Professor Wollny had recorded a number of experiments on this point, and, it being an arbitrary process, he had thought it his duty to follow the manipulation prescribed by Wollny as closely as possible, especially as the requirements were so easy to comply with. In conclusion Mr. Allen said that he was personally extremely sorry that the experiments on the Danish butter had turned out as they had done. He would have much preferred that they should have established the constancy of the composition of butter-fat rather than its variation beyond the accepted range of composition. But public analysts must not rest on their oars, but at once set to work to solve in a satisfactory manner the new problem raised by the experiments in question, and he had no doubt of their ultimate success. They must remember that a dozen year ago, before Messrs. Angell and Hehner devised their well-known process, they had no reliable method of detecting foreign fats in butter at all. He thought they should all combine for the purpose of overcoming the present difficulty, and he therefore proposed "That a Committee, consisting of the President for the time being, Dr. Vieth, Mr. Hehner, Mr. Faber, and himself, with power to add to their number, be appointed to consider and report on the possibility of improving the methods of analysing butter for the detection of adulteration." He relied on Dr. Vieth and Mr. Faber, not only for their great experience in the analysis of butter and other dairy products, but because they were, doubtless, in a position to obtain samples of which they knew the exact history, as regards the number of cows from which they were derived, the method of feeding, and so forth. The motion was seconded by Mr. Stokes, and carried after a short discussion.

(*Conclusion of the Society's Proceedings.*)

ON SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. Atwater.

(*Continued from page* 237.)

Loss by Dissociation and Oxidation of Ammonia.

It is frequently urged that in the determination of nitrogen with soda-lime, ammonia may be dissociated at the temperature at which the combustions are conducted, and that in aspirating air through the tube at the end of the operation some of the ammonia may be burned. The following experiments were made in the hope of getting light upon the conditions under which such loss may thus occur in determinations as ordinarily made.

For this purpose means were taken to test the effects of (1) unusually long exposure of the ammonia to heat, (2) very high heat, and (3) exclusion of air. Ammonia was furnished by ammonium sulphate in which the ammonia had been determined by distillation by the method described by Gooch, two determinations giving respectively 21·11 and 21·14, and averaging 21·13 per cent. of nitrogen. About 0·3 gramme mixed with from 0·5 to 1·0 gramme of oxalic acid previously proven to be free from nitrogen were employed in each of the determinations detailed beyond. The soda-lime, made from 1 part of caustic soda and 2½ of lime, and the method of charging the tube, were as previously described. The method, which is that usually followed in this laboratory, involves filling the tube quite full of soda-lime, so as to avoid large open spaces which become filled with gases and allow long sojourn of ammonia in the tube. Where the long tubes were used, the length of the anterior layer of coarse fragments of soda-lime was increased with the length of the tube, the length of mixture of soda-lime and nitrogenous substance and the charging otherwise remaining the same as in the tubes of ordinary length. The time of combustion, about thirty to thirty-five minutes, was naturally a little less than would usually be the case with animal tissues. The flames were turned off at the close of the combustion before the aspiration with air to wash out the ammonia. In No. 10 (see Table II.) the air was expelled before the combustion, and the ammonia washed out at the end by carbonic acid. In Nos. 11 to 19 the expulsion of air and cleaning out of ammonia were effected by a current of hydrogen, which was delivered by a generator so arranged as to permit easy regulation of the flow. The hydrogen was passed over heated copper foil, to remove the oxygen, before entering the combustion tube.

TABLE II.

Determinations of Nitrogen in Ammonium Sulphate under Different Conditions.

By distillation, 21·11 and 21·14, average 21·13 per cent.

By Soda-lime.

Series.	No.	Length of Tube, c.m.	Heat.	Aspirated with.	Nitrogen found. Per cent.		
					Separate Determinations.	Average.	Range of Variation.
1st	1	42	Medium.	Air.	21·12		
	2	42	„	„	21·10		
	3	35	„	„	21·13	21·12	·03
2nd	4	75	„	„	21·11		
	5	75	„	„	21·12		
	6	75	„	„	21·11		
	7	75	„	„	21·12	21·12	·01
3rd	8	75	High.	„	20·80		
	9	75	„	„	20·86	20·83	·06
4th	10	75	„	Carbonic acid.	20·91		
	11	75	„	Hydrogen and pump.	20·70		
	12	75	„	„ „	20·81		
	13	75	„	„ „	20·84		
	14	75	„	„ „	20·90	20·83	·21
5th	15	75	Medium.	„ „	21·10		
	16	75	„	„ „	21·10		
	17	75	„	„ „	21·12		
	18	75	„	„ „	21·11		
	19	75	„	„ „	21·13	21·11	·03

(*To be continued.*)

HLY RECORD OF GENERAL RESEARCHES INTO ANALYTICAL CHEMISTRY.

IFICATION OF SULPHURIC ACID FOR KJELDAHL'S PROCESS. G. LUNGE. *Zeitschr.* *Chemie*, *No.* 23.—Meldola and Moritz recommend to heat 10 c.c. of the acid two hours with ·05 grm. of potassic nitrite. They take it, therefore, for that the nitrogen contained in commercial acid is always in the form of . The usual impurity is, however, nitrosylsulphuric acid, which the manu- tries to destroy by addition of ammonium sulphate. The decomposition is, never complete, so purified acid generally contains both nitrosylsulphuric acid ionia. The author criticises their method, which may give good results if the of ammonia is known and the theoretical amount of potassic nitrite is added, h fails if any notable excess of nitrite is used. Then it is a great error to that nitrous acid is completely expelled on boiling. Meldola and Moritz have ed the fact that the very stable nitrosylsulphuric acid is formed, which is but volatile. This fact is so well known that further experiments seemed almost us, but to contradict the work of such an analyst as Meldola the author had experiments carried out. A preliminary trial showed that ·2255 grms. of itrite dissolved in 100 c.c. pure sulphuric acid gave a very strong nitric reaction acid had been boiling for two hours. A quantitative experiment was then d; ·392 grms. of sodium nitrite was dissolved in 250 c.c. of pure sulphuric acid sp. gr., and 50 c.c. of this mixture diluted and titrated with permanganate, owed a total of ·1764 nitrous acid. The remaining 200 c.c. were now boiled hours, which caused 100 c.c. to distil over. The residue was now titrated, and a total of ·175 grms. nitrous acid. The greater portion of nitrous acid was still present. A second experiment gave before boiling ·1938, after boiling ms. of nitrous acid. These experiments conclusively show that the purification roposed by Meldola and Moritz is absolutely useless, and only then of avail e quantity of ammonia is accurately known, so as to regulate the quantity of te. But in this case the simplest way is really to deduct this quantity (if not) from any quantity found during an analysis.

L. DE K.

ERMINATION OF NITRATES IN WATER. S. C. HOOKER. *Berichte*, Dec. 10, 1888.—hor makes use of the property of the solution of carbazol in conc. H_2SO_4 of deep green on the addition of small quantities of oxidising agents. The re- delicate enough to detect nitric acid in water containing one 2-millionth part of tance. The following is the method used: A measured quantity of the water, r less, is mixed with 4 ccm. of conc. H_2SO_4, and after cooling, a small quantity ol in conc. H_2SO_4 added. The colouration is compared with that obtained with quantities of KNO_3 solution treated in exactly the same manner. A more publication is promised.

A. L. G.

MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

Right-handed Polarising Honey. Von Lippman. *Zeitschr. f. angew Chemie, No.* 22.—The author confirms the idea of Benseman (see Analyst, 1888), and finds that when in the neighbourhood of sugar-refineries bees swarm there in their thousands, and regale themselves on the sugar. Their honey is then very clear and thin, but almost without the valued aroma. Four samples of such honey, analysed by the author, contained respectively 4·88, 3·92, 16·38, and 9·93 per cent. of saccharose. Samples showing such percentages cannot, therefore, be legally called adulterated. L. de K.

Influence of Different Cattle Foods on the Composition of Butter. A. Mayer. *Landw Vers*, 35.—The author has made a series of experiments to ascertain the composition of butter, and has arrived at the following conclusions:—1. There is a close relation between the specific gravity of butter-fat and the percentage of volatile acids. An increase in the one gives an increase in the other. 2. The melting point of the fat is not much influenced by the tributyrin, as it chiefly depends on the amount of triolein. 3. The amount of volatile acids in the butter of the milk of any particular cow is not so constant as was formerly believed, but is influenced by the quality of the food the cow has been fed on. 4. The amount of volatile acids is gradually increasing during the period of lactation. 5. When the food greatly consists of mangelwurzel the butter shows the highest percentage of volatile acid. Grass and clover will cause a larger amount than ensilage. 6. Hay and ensilage will cause the butter to have a high melting point, whilst green fodder will cause the lowest one. Mangelwurzel gives an intermediate melting point. 7. The solidifying point rises or falls with the melting point. 8. Cattle grazing in the fields will give the best milk and the richest butter. The author finds the quantity of deci-normal soda wanted for the distillate of 5 grms. of butter to vary from 20 to 30 c.c. L. de K.

The Occurrence of Boric Acid in Wine. George Baumert. *Ber.*, Dec. 10th, 1888., p. 3290.—The author's attention was drawn to this subject by his finding boric acid in every sample of a number of Californian wines analysed by him three years ago.[*] Recently, Professor Rising, in San Francisco, stated that boric acid is a characteristic constituent of all Californian wines.[†] Soltsien[‡] found that boric acid is a very frequent constituent of wines, and also occurs in cultivated and wild vines (Ampelopsis quinquefolia) in Saxony, whilst Ripper[§] not only found it in 1,000 samples of German and other wines, but also in various parts of different kinds of vines. In one case he separated it as fluoboride of potassium from 2l. of a Riesling wine. The author has, for three years, tested every German, French, and Spanish wine which came into his hands, and found boric acid in them all; and also in must (fresh juice) from the Freyburg and Naumburg districts. He also systematically examined the growing vines in Thuringia in Saxony, and invariably found boric acid in the leaves, tendrils, wood, grapes, and grape-stems, examining 0·2 to 0·4 grms. of the ash in each case. The same results were obtained on examining a number of leaves and woods from the Freyburg district. A. L. G.

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[*] Nobbe's *Landwirthschaftliche Versuchsstationen*, 33, 39-88.

[†] Report of the Sixth Annual State Viticultural Convention, San Francisco, 1888.

[‡] *Pharm. Zeitung*, 33, No. 40, p. 312; ibid., No. 90.

[§] *Weinban u. Weinhandl*, No. 36, 1888, and Analyst, Dec., 1888.

THE ANALYST.

FEBRUARY, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual Meeting of the Society was held on the 9th ult., at Burlington House, Piccadilly, Mr. A. H. Allen in the chair.

The minutes of the previous meeting were read and confirmed.

The auditor's report on the accounts for the past year was read, and will be printed nd circulated as usual.

Mr. ALLEN then delivered his valedictory address as follows:—

THE PRESIDENT'S ADDRESS.

Following the custom which has prevailed in previous years, I now have to bring efore you a *résumé* of the past work and present position of the Society.

In the first place, it is gratifying to find that the progress of the Society, so far as t is represented by the number of members, is satisfactory. In addition to the resignaion of two associates, three members have been removed from the list for non-payment f arrears of subscription, and six associates for the same reason. To counterbalance his, we have the election of ten new members and four new associates, so that the total umber of subscribers is exactly the same as in the previous year; but the number of rdinary members is increased by four, and that of the associates diminished by the same umber, the honorary members still numbering ten. The Society has also suffered a loss in the death of Dr. William Wallace, of Glasgow. Dr. Wallace was for many years enior member of the firm of Wallace, Tatlock and Clark, and occupied the post of Public Analyst for Glasgow and several neighbouring counties and boroughs. He anked as one of the ablest and best-known chemists in Scotland, and although the istance of his residence from London prevented him from regularly attending our eetings, he always took a great interest in the Society. Personally, I feel in the death f Dr. Wallace the loss of an intimate friend, who was always ready to give me the est advice and assistance in his power.

Not only has the Society maintained its numbers, but the papers read have fully equalled in interest and importance those of previous years. They have numbered 34, as against 30 during the previous twelve months. The following is a list of the papers contributed to the Society, in addition to which there are several papers, such as that of mine on the presence of cocoa-nut oil in a sample of lard, which, though not actually read before the Society, would have been so had the matter arisen during the session:—

PAPERS IN 1888.

Date.		Author.
Jan. 11.	"Laboratory Notes"	A. H. Allen
Feb. 8.	"A Sample of Navy-green Paint"	Bertram Blount
„	"Composition of Milk and Milk Products"	Dr. Vieth
„	"An Instrument for Calculating Milk Results"	H. D. Richmond
„	"An Apparatus for Nesslerising"	A. W. Stokes
March 14.	"Iodine Absorptions, Combining Weights, and Melting-points of certain Fatty Acids"	Rowland Williams
„	"Some Analyses of Yeast"	Rowland Williams
„	"Some Analyses of Goose Fat"	W. C. Young
„	"Pepper Adulteration and Pepper Analysis"	F. W. Rimmington
May 9.	"Some Effects of Food Preservatives on the Action of Diastase"	Dr. Leffmann and W. Bean
„	"Wear and Tear of Platinum Dishes"	Dr. Vieth
„	"Notes on Liquorice and Spanish Juice"	B. Dyer
„	"A New Form of Fat Extraction Apparatus"	Bertram Blount
June 20.	"Polluted Drinking Water and the Closure of Wells	Dr. A. Hill
„	"Adulteration of Lard with Cotton-seed Oil"	A. H. Allen
„	„ „ „	Otto Hehner
„	„ „ „	E. W. T. Jones
„	„ „ „	R. Williams
„	"Temperature of Ether in the Soxhlet Extraction"	Dr. Vieth
„	"Notes on Senna"	C. Heisch
„	"Pepper Analysis"	C. Heisch
July 27.	"On the Use of the Term 'Normal' in Volumetric Analysis"	A. H. Allen
„	"On the Examination and Method of Stating the Results of the Analysis of Boiler-waters"	A. H. Allen
„	"Note on the Examination of Lard"	Thos. S. Gladding
„	"Water Reports—a Dilemma"	M. A. Adams
Nov. 14.	"Determination of the Melting-points of Soap-thickened Oils"	W. F. K. Stock
„	"On Condensed Milk"	J. C. Shenstone
„	"On the Preservation of Milk Samples"	H. D. Richmond
Dec. 12.	"On the Determination of Citric Acid in Lemon-juice"	Rowland Williams
„	"On some Abnormal Samples of Butter"	A. H. Allen
„	"The Werner-Schmidt Method of Determining Fat in Milk and Cream"	A. W. Stokes
„	"On the Examination of Lard for Adulteration"	Thos. S. Gladding
„	"On the Action of Iron Pipes on Water Conveyed therein"	Sidney Harvey

Looking at the immediate objects of the Society, I think we may fairly claim that during the past two years the methods of examining food and drugs have made marked progress. We have been confronted with several novel forms of adulteration, which at first taxed all our resources to discover, but which we may now fairly claim to be in a position to detect with certainty. The use of pepperette, or ground olive-stones, for the adulteration of pepper is one of the cases in question. Although referred to as a possible and probable adulterant of pepper fully ten years ago, it does not seem to have been extensively used until comparatively recently, when pepper adulterated with poivrette made its appearance in various parts of the kingdom, but notably in the neighbourhood of Liverpool, and it was Dr. Campbell Brown, Public Analyst for Liverpool and Lancashire, who first called prominent attention to the question, besides pointing out methods for its detection.

The adulteration of lard has also grown enormously during the past year or two, d, instead of meeting with the large proportions of water formerly so common, lard is w sophisticated in a novel and ingenious manner, namely, by adding a mixture of cotton- d oil and beef or mutton stearin. Mr. E. W. T. Jones appears to have been the first alyst on whose certificate the prosecution for the presence of cotton-seed oil took place, d he was quickly followed by other analysts. For a time the sale of lard adulterated th cotton-seed oil seems to have received a serious check, but knowing how skilfully ch sophistications are conducted, and with the express purpose of baffling the Public alyst, we may expect that those interested are simply seeking a safe method of eluding r vigilance. For a long time it seemed as if the detection of beef-stearin in lard uld only be effected inferentially, and until quite recently I was unable to satisfy yself of the presence of beef-fat in several samples of lard which I strongly suspected contain it. I believe, however, that difficulty is now surmounted, and with the aid some hints from Dr. Campbell Brown I have been able to detect positively the esence of beef-fat in lard. The use of cocoa-nut oil as an adulterant of lard does not em yet to have become very extensive. I have condemned one sample myself on this ound, and have examined another submitted to me from a brother-analyst. But the tection of this adulterant presents no difficulty, and hence the use of cocoa-nut oil is t likely to be very extensively practised.

With respect to butter, the determination of the volatile acids by the Reichert ocess has of late become fairly general, but I find comparatively few analysts have opted the precautions found necessary by Professor Wollny, of Kiel, a translation of ose report and paper, by Mr. O. Hehner, was published in the ANALYST last winter. annot but feel the gravity of the figures I submitted to the Society at its last meeting, owing that the range of variation in the composition of genuine butter was greater than d been generally recognised; but the fact being absolutely beyond dispute it seemed to me at however much we might regret the immediate consequences of the discovery, it s our plain duty to accept the position, and strive so to modify and improve our ethods of analysis as to render it possible to distinguish a butter adulterated with a all percentage of margarine from one which was simply abnormal in composition, and believe it will be found possible to effect this. A committee has been appointed to vestigate the matter. For various reasons, it is not desirable to increase the number the committee to any great extent, but it is to be earnestly hoped that other Public alysts, and the members of the Society generally, will co-operate with the committee, o have undertaken work of a very laborious and responsible character. With their lp and sympathy much may be done, and it is, in my opinion, a great misfortune at the Public Analysts of this country, who have done so much good work in the past, ould have to work under such disadvantageous conditions, and without that encourage- nt from other chemists which they have a right to expect. That the Government of e country should persistently ignore the Public Analysts as a body, as has been done his official position by the Chancellor of the Exchequer, among others, is a lamentable t. The Public Analysts, as a body, are now a set of competent and highly trained n, doing their disagreeable duties without fear or favour, while liable at any time to ve their reputations injured by reference to a Court of Appeal, unsatisfactory in its mposition, and having a narrow and one-sided idea of its duties.

In a few months the position of some of the Public Analysts will be materially anged, as many will hold their appointments under the newly created County Councils. is only to be expected that these bodies will, like other new brooms, sweep clean, ecially as the only argument for supporting some of the candidates seems to be that y are prepared to reduce salaries all round. Under these circumstances it behoves blic Analysts to stand together and make a determined and united effort to resist the attempt, which will doubtless be made in some quarters, to reduce still further the already insufficient remuneration of many of those holding appointments under the Sale of Food and Drugs Act.

As to the future, I think we may, at some time, see an amended Act passed. We are sadly in need of a revised system of reference in the case of disputed analyses. Again, it is very desirable that some means should be devised for tracing milk and butter to their sources, so that if peculiarity of composition be really due to natural causes the vendor may have the fact demonstrated, while, on the other hand, it will become more difficult than at present to plead natural poverty of the milk as an excuse.

The Margarine Act has worked fairly well during its twelve months' enforcement, but experience has shown that it is not free from defects which might be very easily removed by an amending Act.

The difficulty of dealing with drugs is, at present, greatly increased from the absence of any authoritative and legal standard, although it is a curious fact that the Pharmacopœia was not made the formal standard in the Sale of Food and Drugs Act Amendment Act, of 1879, simply because Dr. James Bell held that it was *already practically accepted as the standard.* Unsatisfactory as the existing Pharmacopœia is, and discreditable to certain of those entrusted with its compilation, its formal adoption as the legal standard for drugs mentioned therein would be a distinct advantage, and would have the additional good effect of hastening the publication of a new edition, which might be free from the defects which characterise that of 1885.

There are other directions in which the Adulteration Act might be extended and improved. It certainly should apply to such articles as disinfecting powder, which at present escapes through its meshes; and I think it is not too much to hope that in the future we may see its scope extended so as to make it an offence to sell as "all wool" an article which is in great part cotton or other fibre, and even to prevent the false description of articles of jewellery.

And now it simply remains for me to express my regret that I have on many occasions failed to fill this chair in a satisfactory manner. I am fully aware of my own shortcomings in this respect, but have the satisfaction of knowing that in the immediate future the position lately occupied by me will be filled by one in whose care the dignity and interests of the Society of Public Analysts of Great Britain may be left with the fullest confidence.

Mr. Allen proposed, and it was unanimously resolved, that a vote of thanks be accorded to the Chemical Society for the use of their rooms during the past year.

The election of Officers and Council for the ensuing year was then proceeded with, and on the ballot-papers being opened it was announced that the following gentlemen were elected :—

President.—M. A. Adams, F.R.C.S., F.I.C.

Vice-Presidents (who have filled the office of President).—A. H. Allen; A Dupré, Ph.D., F.R.S.; C. Heisch; Alfred Hill, M.D.; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President).—Sir Chas. Cameron, M.D.; S. Harvey; T. Stevenson, M.D., F.R.C.P.

Treasurer.—C. W. Heaton.

Hon. Secretaries.—Bernard Dyer, B.Sc.; Otto Hehner.

Other Members of Council.—C. E. Cassal; R. H. Davies; W. Fox; R. H. Harland; W. Sedgwick Saunders, M.D.; P. Vieth, Ph.D.; C. R. Alder Wright, D.Sc., F.R.S.

The names of those members of Council whose term of office has not yet expired, and who consequently do not retire this year, are J. Baynes; J. A. R. Newlands; E. Seaton, M.D., F.R.C.P.; W. J. Sykes, M.D.; J. A. Voelcker, Ph.D.

The following gentlemen were also elected:—

As Members: T. S. Gladding, New York; Professor V. Stein, Copenhagen.

As Associate: Sydney Steel, assistant to Mr. Bernard Dyer.

Mr. ALLEN then vacated the chair, which was thereupon taken by the newly elected President—

Mr. ADAMS, who thanked the members for the honour which had been conferred upon him, and said he was fully conscious of the great gap which their retiring President would leave behind him, but he rejoiced to know that Mr. Allen's large and varied experience would not be lost to the Society—they would have him with them in the future, as they had had, so much to their advantage, in the past. He (Mr. Adams) was, however, afraid that it could not be denied that Mr. Allen had left them in somewhat of a mess—a greasy mess. He had certainly upset the fat in the fire, and they would not want for lack of work in the forthcoming season in clearing up after him. Of course they would have him to assist them, and the wonderful instinct that had put them in the right direction, although he had made this mess just at the end of his term, showed that Mr. Allen was just the one to clear it up for them. In conclusion, Mr. Adams said that he could assure them he should have the greatest pleasure in devoting himself to the interests of the Society, and nothing should be wanting on his part to show how fully he appreciated the honourable position to which they had elected him.

The following papers were read:—

"Remarks on a Recent Case of Alleged Injurious Pollution of the River Medway by the Sewage of Maidstone." By Dr. Dupré, F.R.S.

The discussion on this paper was adjourned until the February meeting, in the hope that Dr. Tidy and Mr. Wynter Blyth might then be able to attend.

"On the Analysis of Pepper, and the Occurrence of Piperidine therein." By Dr. W. Johnstone.

"On the Westphal Balance." By Mr. W. F. K. Stock.

The Annual Dinner was afterwards held at the St. James's Hall Restaurant, where a very enjoyable evening was spent by the members and their friends.

The next meeting of the Society will be held at Burlington House, on Wednesday, the 13th inst., when the adjourned discussion on Dr. Dupré's paper will be resumed.

(The papers by Dr. Dupré, Dr. Johnstone, and Mr. Stock, will be published in our next issue.)

ON THE DETERMINATION OF CITRIC ACID IN LEMON-JUICE.

BY ROWLAND WILLIAMS, F.I.C., F.C.S.

(Read at the Meeting, December, 1888.)

THE general chemistry of citric acid has already been so ably dealt with in the excellent and exhaustive papers by R. Warington (*Journal of the Chemical Society*, vol. xxviii., p. 925), and by B. J. Grosjean (*Journal of the Chemical Society*, vol. xliii., p. 331), that it is not necessary for me to dwell at any length on that part of the subject. I may say at once that my special object in bringing this matter before the Society of Public Analysts is to promote a discussion among the members, with the view to attain, if possible, a standard process for estimating the citric acid in lemon-juice, as, in the face of recent improvements in analytical chemistry, this question certainly deserves further consideration—former methods being more or less unreliable, owing to the unsatisfactory nature of the indicators employed.

Litmus, logwood, and cochineal solutions, all of which have, I believe, been used in the past, are unsuitable indicators, while litmus-paper is, for a reason which will presently appear, not very much better. Samples of lemon-juice are sometimes sent to my laboratory for examination by calico-printers and others, and my results nearly always come out lower than those of Mr. G. H. Ogston, upon whose certificate, lemon-juice is, I understand, generally sold in this country.

These discrepancies having occurred rather frequently, I communicated with Mr. Ogston, in order, if possible, to come to an understanding as to the method to be adopted in the analysis of future samples, so as to avoid any further differences between our results.

In the course of his reply Mr. Ogston said, "The process agreed upon for the commercial analysis of lemon-juice is titration by carbonate of soda solution, using litmus-paper as indicator."

Mr. Ogston then points out certain precautions necessary to be observed, and admits that the exact point at which to cease adding the carbonate of soda is difficult to see, and can be determined only after a good deal of experience.

I was rather surprised to hear of an analyst employing carbonate of soda in estimations of citric acid. I myself use pure caustic soda solution, which I find to be preferable in every respect.

Litmus-paper is also, in my opinion, a very unsatisfactory indicator, as it is affected to a considerable extent by normal citrate of soda—the real end reaction being thus difficult to tell with accuracy.

It is a well-known fact that citrate of soda solution, according to its degree of dilution, turns pink litmus-paper more or less blue, and it becomes, therefore, to a certain extent a matter of chance as to what excess of soda must be added when litmus-paper is used as an indicator.

In order to overcome this difficulty it is, I believe, customary to ascertain the exact strength of the alkali by means of pure citric acid and pale litmus-paper, taking as nearly as possible the same shade of blue for the end reaction, both in standardising and in performing the actual analysis; but even using every precaution, I find this plan to be unsatisfactory, in addition to being very tedious.

In estimating citric acid I have long since discarded litmus as an indicator in favour of phenolphthalein, as recommended by R. T. Thomson (*Journal of the Society of Chemical Industry*, vol. vi., p. 195) and other chemists. This latter indicator I have proved to be quite unaffected by citrate of soda, whereas this salt is distinctly alkaline to litmus, as already mentioned. The end of the reaction is also seen with the greatest precision when phenolphthalein is used, one drop of normal caustic soda solution in excess being sufficient to produce an unmistakable change of colour. No reliance can, of course, be placed on results obtained by titrating with either hydrate or carbonate of sodium in the presence of mineral acids, but in their absence neutralisation with alkali gives a fairly accurate idea of the amount of citric acid present, especially in concentrated lemon-juice.

In the presence of other acids which might interfere with the ordinary soda test it is necessary to employ the calcium chloride process described in Mr. Warington's paper.

The plan which I adopt in the analysis of concentrated lemon-juice is as follows: The specific gravity is first taken at 60° Fah. For this purpose some analysts employ a special hydrometer, termed a "citrometer," on the scale of which every ·004 above unity is equivalent to one degree.

As is the case with so many commercial products, the density indication is of little or no value, unless the lemon-juice is free from adulteration. Nevertheless, it is customary to state the "citrometer" indication on commercial certificates. Indeed, many calico-printers in my district actually buy according to the "citrometer" degrees, without paying much attention to the proportion of citric acid present, and thus become ready victims to the wiles of unscrupulous drysalters. In order to determine the total acidity of the juice, I dilute 1,000 grains measure of the sample to 10,000 grains with water in a stopped measuring-flask, and take one-tenth of the whole (equivalent to 100 grains of the original juice) for titration with alkali. This aliquot portion is placed in a large porcelain basin, and diluted with water until the liquid is of a pale yellow shade, and after the addition of a few drops of alcoholic phenolphthalein solution, normal caustic soda solution is run in from a burette until a rose colour appears. In order to avoid any possible chance of error, I always perform the entire process in duplicate. Two determinations of the total acidity should agree within two grains of normal soda solution. The results are calculated into ounces of citric acid per gallon.

As a confirmatory test, another carefully measured portion of the sample may be taken, and without any previous dilution, neutralised with the requisite amount of caustic soda. Rather more than the calculated quantity of a 20 per cent. solution of calcium chloride is next added, and the whole heated in a salt bath for some time.

The calcium citrate is filtered off, washed slightly with boiling water, the filtrate concentrated to a small bulk, taking care to keep the solution neutral, and again filtered. If necessary, the filtrate is again concentrated, and any precipitate which comes down filtered off through a very small filter. The precipitates are gently ignited in a platinum crucible, in order to convert the calcium citrate into calcium carbonate, which is then decomposed by excess of normal hydrochloric acid, filtered and titrated back with normal caustic soda.

When testing genuine samples of lemon-juice I have generally found this process to give figures agreeing fairly well with those obtained by direct titration with soda, the results being in most cases rather lower, owing to part of the total acidity being due to organic acids, other than citric, which do not form an insoluble lime salt.

I have tried the carbonate of soda method used by Mr. Ogston, in comparison with my ordinary process, on several samples of lemon-juice, some of which were taken from pipes imported from abroad, while the others were prepared from lemons pressed and concentrated in my own laboratory. The figures obtained will be found in the accompanying table:—

Ounces of Citric Acid per Gallon.

	NaHO Method.	Na_2CO_3 Method.	$CaCl_2$ Method.
Foreign Samples.			
No. 1	68·24	69·42	64·12
No. 2	63·48	64·56	58·98
No. 3	61·80	62·55	58·24
No. 4	62·88	63·96	60·14
No. 5	60·86	61·84	57·98
No. 6	66·74	67·88	64·92
Laboratory Samples.			
No. 1	94·32	94·56	94·08
No. 2	63·36	64·32	62·52
No. 3	75·48	76·20	75·60

These experiments show plainly that titration with carbonate of soda and litmus-paper invariably gives higher results than titration with caustic soda and phenolphthalein. I may say, in passing, that my carbonate of soda results, although so much higher than the caustic soda figures, are lower than those obtained by Mr. Ogston in testing duplicate samples.

Pure Citric Acid.—We now come to the consideration of the respective values of normal carbonate and caustic soda solutions, when titrated against pure citric acid, as upon this the value of the process entirely depends. For this purpose I obtained samples of citric acid from seven different manufacturers. Very accurately weighed quantities of each were carefully titrated with pure normal caustic and carbonate of soda, using alcoholic solution of phenolphthalein and litmus-paper as the respective indicators. All the experiments were done in duplicate. In the following table are given the number of grains of the seven samples respectively, neutralised by one hundred grains of normal caustic and carbonate of soda solutions:—

	NaHO.	Na_2CO_3.
Sample No. 1	7·03	7·32
„ No. 2	6·96	7·37
„ No. 3	6·94	7·12
„ No. 4	6·99	7·13
„ No. 5	7·09	7·26
„ No. 6	7·14	7·24
„ No. 7	7·02	7·21

On averaging these figures, it will be seen that 100 grains normal caustic soda equal 7 02 grains citric acid, while 100 grains normal carbonate of soda equal 7·23 grains citric acid. For all practical purposes, the 7·02 grains may be regarded as 7 grains, the slight difference being, no doubt, due to the presence of traces of impurities in some of the samples of citric acid. This result would then agree exactly with the figure demanded by theory.

The accuracy of titrations made with caustic soda, using phenolphthalein as indicator, having thus been proved beyond doubt in the case of citric acid, it seems undesirable to employ carbonate of soda in the analysis of lemon-juice, as the estimation then occupies so much longer time, besides not giving such reliable results. I would suggest, therefore, that the citric acid in lemon-juice should in future be determined by titration with caustic soda and phenolphthalein in the absence of interfering bodies, the result to be confirmed, if necessary, by the application of the calcium chloride test previously described.

In conclusion, I should like to add that the calcium chloride method indicated 99·4 per cent. of citric acid, when tried on a portion taken from the mixture of the seven samples of citric acid already referred to.

DISCUSSION.

The PRESIDENT said that he was glad to find that Mr. Rowland Williams appreciated the use of phenolphthalein as an indicator. He thought that the tendency was to use phenolphthalein more and more in all cases in which it was suited for the purpose—that is, where a weak acid was to be determined and the absence of carbonic acid could be ensured. He had no doubt that the process of titrating citric acid and lemon-juice by caustic alkali and phenolphthalein was novel so far as Mr. Williams was concerned; but it had been in constant use for some years, to his (the President's) knowledge, in several laboratories where such essays were required, and the process was fully described in his "Commercial Organic Analysis," vol. i. Phenolphthalein always gave very satisfactory results where the colour could be seen; but in the case of very dark juices it sometimes became necessary to have an outside indicator, in which case very delicate litmus-paper, made by brushing a neutral solution of litmus on to white writing-paper, not absorbent paper, was the best substitute for it. Congo-red paper would probably also be valuable. It was disheartening to learn that chemists of repute were still content to titrate lemon-juice with an alkaline carbonate and litmus. He was not surprised that Mr. Williams's results did not agree with those who employed so antiquated and unsatisfactory a process, and there could be no doubt on which side the truth lay. In titrating with phenolphthalein there was one direction in which he thought an improvement might be made, and that was to use a standard solution of baryta instead of caustic soda. Baryta had the advantage that it was certain to be free from carbonic acid, which, of course, was not the case with caustic soda. However, he would have occasion to refer at greater length to this point later in the evening.

Mr. DYER said he happened to know that Mr. Ogston used litmus-paper that was not bibulous, but painted on one side, and that he laid great stress on that.

Mr. WILLIAMS, in reply, said he had not tried congo-red paper, but congo-red solution did not answer the purpose at all.

THE WERNER-SCHMID METHOD OF DETERMINING FAT IN MILK AND CREAM.

By A. W. STOKES, F.I.C., F.C.S.

(*Read at Meeting December*, 1888.)

Of the various methods of determining fat that I have practically tried, including those of Wanklyn, the Lactobutyrometer, Adams's paper-coil, and the Lactocrite, not one is perfectly satisfactory.

The two former are not accurate, the paper-coil takes too long, and the lactocrite is too expensive and unsuitable for a laboratory.

All who have many samples of milk to analyse will, I think, welcome a method that combines almost the simplicity of the Wanklyn process with the accuracy of the

Adams's paper-coil method, and in addition gives a quickness that neither can pretend to. All this, I believe, the Werner-Schmid method does.

About two months ago, in vol. xxvii., part 4, of the *Zeitschrift fur Analytsche Chemie*, appeared a notice which was translated into the *Chemical News* of October 19th, 1888, and has since appeared in the December number of the *Journal of the Chemical Society*. It is so short that I quote the whole of it:—

> "Dr. Werner-Schmid takes a test-tube of about 50 c.c. capacity, graduated in tenths of a c.c., introduces 5 c.c. of cream or 10 c.c. of milk, accurately measured, adds 10 c.c. of strong hydrochloric acid, boils, with shaking, until the liquid turns dark brown, cools by placing the tube in cold water, adds 30 c.c. of ether, shakes round, lets stand, measures the volume of the ethereal solution, draws off 10 c.c. with a pipette, evaporates down in a weighed porcelain capsule on the water-bath, and finally in an air-bath at 100°. He then weighs and calculates for the original quantity of the ethereal solution. If the process has been rightly conducted, the ether separates from the aqueous solution clear, without the slightest turbidity. The ethereal solution, as it flows out of the pipette, should not show any watery drops. The results are perfectly accurate, and differ from each other and from the ordinary gravimetric methods by less than one-tenth per cent. The operation requires at most fifteen minutes."

Nowhere could I find any further information or any experimental proofs of the accuracy of the method; I therefore now supply my experience. At first I tried to simplify the process by using a 50 c.c. flask, in which it was easier to boil the mixed acid and milk than in a tube. From this, after filling up with ether, I took 10 c.c. for evaporation, assuming that there was present 30 c.c. of ethereal solution of fat. This gave, however, too high results, because some of the ether was taken up by the acid mixture. I now, therefore, use calibrated test-tubes, but do not actually boil the mixture of milk and acid, but plunge the corked tubes into the boiling water of the water-bath for 5 to 10 minutes, then cool them down by immersion in cold water. On adding ether up to the 50 c.c. mark, and vigorously shaking for half a minute, a contraction of the ether takes place; it usually stands at 49·5, while the acid and milk mixture takes up some ether, so as usually to occupy 26 c.c., leaving 23·5 c.c. of ethereal fat solution. After standing for 3 to 5 minutes, this ethereal solution separates completely; 10 c.c. or any aliquot part may be pipetted off and evaporated. I find that flat-bottomed glass dishes, of 2½ in. diameter, are very suitable for rapid evaporation of the portion of ether taken. The whole operation takes from 15 to 20 minutes, but of course a number can be going on at the same time.

Ordinary milks gave the following comparative results by the various methods of—

	Wanklyn.	Adams.	Schmid.	Calculation.
No. 1	2·64	2·91	2·90	3·14
No. 2	2·53	2·71	2·70	3·11
No. 3	..	2·48	2·37	2·95
No. 4	2·04	..	2·53	2·70
No. 5	2·71	..	3·08	..
No. 6	..	..	2·88	3·22
No. 7	..	..	3·48	3 61

These are a few examples of milks received in the ordinary course from various parishes. Most of them are watered samples, taken from the top of the churn, hence the agreement between the Adams or Schmid with the calculated fat is not so near as usual. It is, however, evident that the Wanklyn process does not extract the whole of the fat, even when, as in some of the cases, the milk after weighing was curdled with acetic acid, and its solids boiled with benzoline for two days. The Adams's process and the Schmid agree, I think, within the limits of experimental error.

Skimmed milks gave the following comparative results:—

	Wanklyn.	Adams.	Schmid.	Calculation.
No. 8	0·20	0·30	0·24	..
No. 9	..	0·51	0·50	..
No. 10	..	0·65	0·64	..
No. 11	..	..	1·14	1·24
No. 12	..	..	1·02	1·12

Condensed milks, I find, are best treated by weighing out 10 grms., and diluting this to 100 c.c. with water, then taking 10 c.c. of this for the Schmid process.

A sample gave by Adams's method 12·4, by Schmid's 12·1 per cent. fat.

Stale milks are readily treated by this process. When shaking such a milk to obtain a fair sample an enormous mass of gas-bubbles is formed, such as prevent an accurate volume of the milk being taken. If, however, after shaking vigorously, the milk be poured into a 100 c.c. flask, in which 75 c.c. of water are present, with frequent agitation while pouring, all the bubbles will break, and the flask may readily be filled to the 100 c.c. mark. From this four-times-diluted mixture of the stale milk 10 c.c. may be taken for the Schmid process, and be treated as described:—

No. 13 gave 3·69 when fresh, and 3·63 after an interval of 39 days.
No. 14 „ 0·69 „ 0·66 „ 39 „
No. 15 „ 2·87 „ 2·82 „ 40 „
No. 16 „ 3·12 „ 3·24 „ 17 „
No. 17 „ 2·73 „ 2·76 „ 15 „

Creams.—I find it best to weigh out 10 grms. of the sample, and to make this up to 100 c.c. with water, then to take 10 c.c. of the mixture for the Schmid method. Otherwise, if the cream is treated direct, or only slightly diluted, the pipette cannot deliver its proper amount. Besides this, the resulting ethereal solution is so dense as to make it necessary to wait a long time for the other parts of the mixture to settle after shaking. Samples thus treated gave the following results:—

	Wanklyn.	Adams.	Schmid.
No. 18	42·8	..	42·4
No. 19	..	52·8	52·5

The action of the process is that the acid destroys the casein almost entirely, and acts upon the milk-sugar to produce the brown colour, while the fat is set free and rises to the surface. The ether dissolves this fat, but I think not entirely. There is always a minute amount of fat present in the hydrochloric acid solution. I am trying to find whether this is a constant proportion, so that it might be allowed for.

It is necessary to use ether that has been washed with water, otherwise the alcohol usually present in commercial samples will interfere with the results. No contraction or alteration of volume occurs in the mixture of acid and milk after boiling and cooling, but when the tube is filled to the mark with ether and well shaken a contraction of the total quantity takes place.

I determine the amount of this (usually 0·5 to 1 c.c.) for each tube, and make a mark above the usual 50 c.c. contents-mark, and fill with ether to this before shaking.

The ethereal solution of fat when pipetted off is always acid. I have, however, carefully tested a large number of such combined extracts for milk-sugar, but have never found a trace.

Should the tube, by accident, be filled above its proper mark with ether, it is easy to evaporate off the excess. Should the first portion of ethereal solution be lost, another portion may be readily pipetted off. Doubting whether it was sufficient to shake the mixed fluids for half a minute, and to let stand for 5 minutes only, I have shaken duplicates for longer periods, and have let them stand for 2½ days. The results have been the same. Frequent repetitions of the same milk agree.

It is necessary to see that pipette, burette, and tube are properly graduated and to weigh accurately.

So far the method seems to me to possess the advantages of accuracy, speed, simplicity, cheapness of apparatus, and little loss of reagents. Calibrated tubes, made for the purpose, may be obtained of Messrs. Cetti, Holborn.

EXAMINATION OF LARD FOR ADULTERATION.

By Thomas S. Gladding, New York.

(*Read at Meeting, December*, 1888.)

In the examination of lard the following tests have been found most reliable:—

(1) Specific gravity at 100° C. (2) Hübl's Iodine test. (3) Milliau's modification of Bechi's test. (4) Dalican's "Titre" test. (5) Belden's microscopic test for beef fat, as described by Professor Sharples in a late number of the Analyst. No lard should ever be condemned by a single test. A certificate should never be given except one based upon a full examination.

The "titre" is Dalican's crystallising points of the fatty acids. The term is a very convenient abbreviation, and the value of the test itself will appear from the results of the work done.

The following table gives the tests of eight samples of whose purity no doubt is entertained. Samples 4, 5, 7 were tried in our laboratory. Samples 1, 2, 6, 8 were received from reliable houses, and their purity guaranteed. Most of the work was done in careful duplicate. Hübl's method was strictly followed, the hyposulphate solution being standardised by chemically pure iodine prepared by ourselves, and also by chemically pure iron ammonia sulphate.

	Specific Gravity at 100° C.	"Titre."	Iodine Test.
1. Lard oil	—	31·40° C.	74·60 per cent.
2. Lard (very soft)	0·8610	36·40	68·40
3. Lard	0·8614	36·40	67·60
4. Leaf lard, 1 (soft)	0·8610	38·20	65·80
5. Leaf lard, 2 (hard)	—	41·40	57·00
6. Lard stearine	—	43·50	49·50
7. Beef fat (soft yellow) ...	—	41·60	43·80
8. Cotton-seed oil (summer white)	0·8690	33·30	108·00

The striking point in the above table is the wide range of the iodine test on samples of lard of undoubted purity. The highest tests hitherto published have been about 61 to 62 per cent. With soft lards the figures 67·60 and 68·40 were obtained. The above "titre" tests at once explain this great range of the iodine tests. From Dalican's table we find that a tallow having a titre of 41·40° C. contains approximately 59 per cent. of olein, and one of 43·50 contains 53 per cent. of olein. The iodine absorbed will vary approximately with these varying proportions of olein. In any examination, therefore, of lard the titre of the sample should be known, in order to interpret the meaning of the iodine test. The wide difference between the titre 31·40 of lard oil and 43·50 of lard stearin and the corresponding iodine tests of 74·60 and 49·40 is a further and more extreme illustration of the same truth. The beef fat in the table was a soft yellow fat of lower titre than usual. The common titre of prime solid beef fat is about 44, and would show an iodine test of about 40, the number usually given for beef. Comparing the iodine test 43·80 of beef fat above with the iodine test of 56·20 of lard of about the same titre, it becomes apparent that there is some difference in the chemical nature of the two fats which is demonstrated by the iodine test, but not revealed by the titre. I have been informed by good authority that the cotton-seed oil (summer white) in the above table is the form commonly used for adulteration of lard, and not the cotton-seed stearin as has been stated. The low titre of 33·30 is therefore interesting, and its influence will appear in some analyses to be given later. I have made thorough trial of Belden's microscopic test, and have been greatly pleased with it. It is very valuable for the detection of beef fat in lard, and in most cases is the ONLY reliable test for that adulterant, but requires much practice with samples of known purity and samples of known percentage of adulteration.

The following analyses of samples sent in for examination will prove interesting. Four samples were sent in to determine the chemist's ability to detect adulteration. One sample was to be pure lard, another adulterated, and the other two were to be the products of rival houses that were suspected of adulterating their goods. The samples were numbered 1 to 4, with no other mark.

	1	2	3	4
Specific gravity at 100° C.	0·8610	0·8606	0·8636	0·8631
Titre	36·40	39·60	36·15	36·60
Iodine test	68·40	58·33	76·60	75·40
Belden test	Lard.	Beef lard.	Beef lard.	Beef lard.
Bechi-Milliau test ..	Negative.	Negative.	Strong darkening.	Strong darkening.

Nos. 3 and 4 are at once seen to contain cotton-seed oil from sp. gr., from iodine test, and from Bechi-Milliau test. Sample 2 would be called pure lard, but the crystals of beef fat were formed so readily and abundantly as to show at least 20 per cent. of that adulterant. The impossibility of detecting beef fat without Belden's test is at once evident.

The very low titres of 3 and 4 need explanation. These samples contain at least 25 per cent. of beef fat. The titre should, therefore, be at least 38. The explanation is found in the presence of cotton-seed oil, similar to that in the table, having a low titre of about 33, by which the titre of the mixture is correspondingly lowered. The approximate percentages of cotton-seed oil can only be deduced from specific gravity. Taking that of normal average lard as 0·861, and that of cotton-seed oil as 0·869, we conclude there must be about 32·5 per cent. and 26 per cent. of cotton-seed oil respectively in these two samples.

Two other suspected samples have just given the following results :—

	1	2
Specific gravity at 100° C... ..	0·8600	0·8606
Titre	38·00	38·80
Iodine test	65·40	60·27
Bechi-Milliau test	Blackening.	Negative.
Belden test	Much beef fat.	Much beef fat.

Judged by special gravity and by iodine test, both samples would be pronounced pure. The Bechi-Milliau test in sample No. 1 finds marked confirmation in the fact that though at least 20 per cent. beef fat is present, yet the iodine test, instead of dropping to at least 60, as we should expect from the reducing action of the beef, remains as high as is ever found in a pure lard showing a titre of 38° C. The presence of 5 to 10 per cent. cotton-seed oil can be positively affirmed in view of the whole analysis. Ten per cent. of cotton-seed oil might not have any visible effect on specific gravity, but would raise the iodine test from 65·4 to 70, which in turn is reduced in the above sample to 65·40 by the presence of, say, 20 per cent. of beef fat.

The fatty acids are prepared by saponifying in such a way as to secure as colourless fat acids as possible. They are washed in a separating bulb with several additions of very hot water, and finally filtered through a dry filter paper into a test tube of 1½ to 2 centimetres diameter. The "titre" is taken with a delicate thermometer graduated to tenths of a degree. 5 c.c. are then tested for cotton-seed oil by Bechi-Milliau test, and the remainder may be used to ascertain the combining number. A portion also can be used to obtain the iodine number. The iodine absorbed by the fat acids, multiplied by 95·5 (in the case of fats consisting of stearin, palmitin, and olein), will give the iodine absorbed by the original oil.

The great advantage of Dalican's method over any other method of finding the melting-points of oils is the accuracy of the results obtained. The results are correct, as proved by scores of duplicate tests to within ·1, or at most ·2 degrees Centigrade.

While the above work seems to destroy the value of Hübl's iodine test for examining lard (by itself), yet it by no means cripples the analyst in his ability to reach a correct conclusion when the examination is made a complete one, and all the data attainable have been ascertained.

ON THE ACTION OF IRON PIPES (RUSTED) UPON WATER CONVEYED THEREIN.

By S. Harvey, F.C.S., F.I.C.

Read at Meeting, December, 1888.

That iron-oxide effects some changes upon certain constituents of drinking water, specially as regards the nitrates present, has long been known, and I lay claim to no originality in bringing the following observations before the Society of Public Analysts.

Nine months ago (March) the Public Water-supply of the Borough of Ramsgate was extended to the village of Minster, the latter place having previously derived its water for drinking purposes from private wells, most of which were polluted.

No sooner however had the Ramsgate supply reached Minster than complaints were made as to its character and appearance. It was very turbid, depositing ferruginous sediment, and when submitted to analysis was found to yield in addition to a considerable amount of combined chlorine "high figures" for "*Ammonia,*" "*Oxygen absorbed,*" and "*Nitrites,*" and this state of things has now continued for several months.

Samples taken from the new supply at Minster were submitted to me, and I had some difficulty in convincing the senders that the appearance of the water was not due to sewage or organic impurity.

As my acquaintance with the Ramsgate water has extended over several years, and as the supply is very pure and uniform, ranking among first-class waters, I felt that a good opportunity presented itself for testing the action of iron pipes (presumably rusted) upon water of a known constant composition. Accordingly I had a number of samples taken all at one date, July 23rd last, and with as little interval between each as the distance would allow. Every care was used both in the collection of the samples and the analysis, which followed immediately. In the case of the *turbid* samples the clear supernatant water only was analysed.

None of these samples contained sufficient iron in *solution* to affect "ammonium sulphide solution."

Samples 1, 2, and 3 were taken from the old Ramsgate mains. Samples 4, 5, and taken at Minster after a run of from five to six miles through the new mains to er place, the pipes being four, five, and six inches in diameter.

PUBLIC WATER SUPPLY, RAMSGATE.

	Taken at Ramsgate (old mains).			Taken at Minster (new mains).		
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
ance	clear	clear	clear	clear	very turbid	turbid
ed Chlorine	5·60	5·60	5·25	5·53	5·88	5·60
en as Nitrates Nitrites	0·72	0·74	0·72	0·19	0·49	0·58
nia	trace	trace	trace	0·0182	0·0168	0·0025
inoid NH_3	0·0017	0·0017	0·0018	0·0016	0·0014	0·0022
in 4 hours	·024	·024	·028	·090	·118	·068
Solids	35·70	35·63	36·19	29·89	34·79	34·72
t	very slight	very slight	slight	slight	Iron-oxide	Iron-oxide
s	none	none	none	in quantity	in quantity	in quantity
Nitrogen in Nitrates and Nitrites	0·720	0·740	0·720	0·205	0·504	0·582
en Missing	—	—	—	0·522	0·223	0·145

.B.—The figure for combined chlorine in the Ramsgate water fluctuates a little, fers but little from what it was five years ago.

mitting details common to both sets of samples, it will be observed that the s have diminished considerably in the last set (4, 5, and 6). The ammonia has rom traces in the first set to considerable amount in the latter, while nitrites in quantity in the latter, their amount being measured *practically* (minus a small on) by the oxygen absorbed.

wo questions present themselves. What has become of the missing nitrogen in ree last samples, and how may the small but distinct variations of combined e be accounted for?

here is also a moral consequent upon the above, which was the chief reason for my g this matter before the Society.

ow far is an analyst justified in pronouncing an opinion upon the purity of a g water in the absence of *all* information as regards the *origin* and mode of and conveyance of the same, or when such particulars are purposely witheld?

t would be interesting to know how the Minster supply would fare in the hands nalyst who had been kept in ignorance of its antecedents.

n connection with this subject, viz., the effects of iron oxide upon water, perhaps llowing analyses may be interesting, and they confirm some remarks by Dr. E. and (vide *Water Analysis* p. 19).

he water from these wells has been a cause of unpleasantness between analysts own experience, very pure as it is.

Water from "Lower Tertiaries," (Thanet Beds) below "London Clay."
Taken from wells six miles N.N.W. of Canterbury.

	No. 1	No. 2
Appearance	very turbid	rather turbid
Combined Chlorine	7·14	7·14
Phosphates	very distinct traces	distinct traces
Nitrogen as Nitrates	0·02	0·02
Ammonia	0·1190*	0·0878*
Albuminoid Ammonia	0·0014	0·0019
Oxygen absorbed in 4 hours	·023	0·026
Total Solids	52·78	52·22
Deposit	chiefly Ferruginous	chiefly Ferruginous
Sulphates	present	present
Nitrites	absent	absent

Canterbury, December 10, 1888. (*Conclusion of the Society's Proceedings.*)

NATURAL APERIENT BITTER WATERS.

By Otto Hehner.

Some time ago the attention of the medical profession was drawn to the important subject of accuracy in dispensing of medicines, by the publication of a joint report by Dr. Edward Seaton and myself. That report having been productive of some good, I venture now to refer to an allied subject, in the hope that a remedy may be found for an evil which concerns medical men and the public alike.

For a number of years past the sale of natural aperient magnesian waters has been considerably increasing, and there is now a large variety of such waters at the disposal of the practitioner. These waters are generally sold in bottles, upon the labels of which, in most cases, analytical figures are printed, which allege to represent the composition of the contents, and are doubtless intended as a guide in fixing the dose of the water to be used. There are also, generally, directions on the bottles to the effect that a wineglassful is the average dose.

It should be generally known that the waters in question, being obtained in the majority of cases from very shallow wells, are liable to very wide fluctuations in composition and consequently in therapeutical activity, and that it is impossible to represent, once for all, the composition of the waters by a single analysis. True mineral waters, coming from great depths, may and do remain unchanged in composition for a long number of years, but magnesian bitter waters, as at present collected, fluctuate in strength almost from day to day; stronger waters collect at the bottom of the wells than in the upper strata, and it is possible, therefore, to obtain at will strongly or relatively slightly saline solutions from the same well.

The following analyses, obtained from samples which I have lately purchased in the London market, represent six different waters, all coming from the Ofen district in Hungary, and frequently taken to be equivalent of each other.

10,000 parts contained:—

	A	B	C	D	E	F	G
Sulphuric acid	187·56	197·08	232·20	242·34	247·48	279·37	310·60
Chlorine	13·80	15·95	8·87	10·60	10·30	13·85	14·00
Carbonic acid	2·29	2·51	3·87	4·36	3·08	5·81	5·06
Magnesia	54·00	53·19	56·85	71·95	65·66	97·24	101·60
Lime	6·52	6·12	6·36	6·42	6·24	7·22	7·27
Soda	67·75	79·36	91·74	87·88	96·40	91·05	92·17
Specific gravity	1031·1	1032·7	1037·0	1039·5	1039·7	1048·7	1049·6

Samples D and G represent the same brand of water at different times.

To render these analyses more readily comparable with each other, I will assume that the whole of the magnesia and soda is present in the form of sulphates, which may the more readily be done as the proportions of carbonate of magnesia and chloride of sodium are small, and but little different in the seven samples :—

Magnesium sulphate ..	162·00	159·57	170·55	215·85	196·98	291·72	304·80
Sodium sulphate ..	155·16	181·76	210·11	201·27	220·79	208·53	211·09
	317·16	341·33	380·66	417·12	417·77	500·25	515·89

Taking the proportion of magnesium sulphate as the standard of strength, it is seen that of sample B 1·91 parts are necessary to produce the same effect as one part of sample G.

As the laxative powers of magnesium sulphate and sodium sulphate are nearly equal, we may more fairly compare the totals of the two salts as the measure of the activity of the waters. In that case, 1·63 parts of sample A are equal in effect to one part of G.

The readiest measure of the strength is the specific gravity. According to this A stands to G as 1 to 1·59, the quantities to be taken being of course inversely proportional.

It appears to me that the medical profession should be distinctly aware of the wide differences in the composition of these waters, and should either insist upon some measure of uniformity, which by care in bottling the waters could easily be obtained, or upon an exact statement of strength upon each bottle, be it by analytical figures, or by specific gravity only.

Imagine a patient who has been accustomed to a particular dose of, say, water A, taking an equal dose of sample G. The purgative action would be increased by almost two-thirds. Even if serious consequences might not be the result, the uncertainty of the remedy is apparent.

There could not be any practical difficulty whatever to obtain uniformity in composition, by excluding from the wells all surface water, or such coming from strata poor in saline ingredients. Even then fluctuations, but within narrower limits, would occur. An exact statement of the specific gravity of the contents of each bottle should therefore be made, so that any medical man would be in a position exactly to determine the necessary dose.* The analyses at present given upon the labels are of no value whatever for this purpose, as they do not represent the composition of the contents, but of samples specially selected to show high saline proportions, and often made many years ago.

A NEW PEPPER ADULTERANT.

By F. Wallis Stoddart, Public Analyst for Bristol.

A new material in extensive use for the adulteration of pepper having recently come under my notice, I wish to call the attention of public analysts to it, that a united effort in all quarters may stamp out what bids fair to become a very successful fraud.

The material in question consists of rice starch, barytes, calcic carbonate, and lead chromate, all, of course, finely ground and intimately mixed. The lead chromate amounts to about 10 per cent. of the whole compound. By the addition of about 5 per cent. of this mixture, the colour of pepper is so immensely improved as to raise

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[*] The figures of the specific gravity above 1,000 very nearly representing the sum of the magnesium and sodium sulphates per 1,000 parts of the water. Thus in sample A a specific gravity of 1031·1 corresponds to 31·7 parts of the sulphates per 1,000 of the water.

its market value very considerably, but I have met with pepper containing fully 10 per cent.

Such confidence do the manufacturers of this delectable mixture feel in it, that they guarantee their peppers coloured with it to be genuine, and I saw one invoice endorsed, "warranted genuine as per Somerset House analysis."

Nevertheless the detection of this adulteration is not difficult. The increased ash and presence of rice would arrest attention, whilst the peculiar and unnatural tinge communicated by the chromate is readily noticeable. The method of examination, however, which has proved most successful in my hands is agitation with chloroform, by which means the mineral ingredients of the adulterant are obtained in their natural combinations.

The well-washed residue is gently warmed until the chloroform has evaporated, treated with a very little sodic carbonate solution and allowed to cool. A few drops of ether, which has been agitated with aqueous hydrogen peroxide, are then added, and the mixture carefully acidified with hydrochloric acid, when the delicate blue colouration is readily obtained, and the barium and lead remain to be estimated in the usual way.

MONTHLY RECORD OF GENERAL RESEARCHES INTO ANALYTICAL CHEMISTRY.

Estimation of Starch for Technical Purposes. H. Schreib. *Zeitschr f. angew Chemie, No.* 24, 1888.—The author recommends to estimate the proteids by combustion, and also to take the moisture and the ash. The starch is thus found by difference, but an allowance is made for other carbo-hydrates and fatty matter. Although it seems at first sight absurd to make three determinations instead of one, in practice this way will be found more convenient than a direct estimation of the starch. The best direct process as yet known, viz., Lintner's, takes more than 6 hours, in which time it is much easier to finish the combustion and do the water and ash. Even if Lintner's process left nothing to be desired from a point of accuracy, it will still give unreliable results to the starch manufacturer, because no notice is taken of the starch, which will be retained by the albumenous matter. The author finds that every pound of gluten will cause the loss of one pound of starch. Soluble proteids will, of course, cause no loss. The value of a commercial *starch* is best got at by direct experiment. Four parts of starch boiled with fifty parts of water must give a proper set paste. Before boiling the starch must be powdered and thoroughly mixed with the water. L. de K.

Impure Reagents. L. L. de Koninck. *Zeitschr. f. angew. Chemie, No.* 1, 1889.—Lead peroxide often contains manganic peroxide. Being sometimes used for the detection of traces of manganese, it is important to know whether it is perfectly free from that body. Mere boiling with nitric acid will not be always successful. The best way is to first heat a portion of the powder with strong sulphuric acid to dryness, which will yield plumbic and manganous sulphates. A fresh quantity of the lead is now added and the whole boiled with nitric acid, when the test will be successful. Ether often deserves the name of *sulphuric* ether, as it sometimes actually contains free sulphur. This impurity will be easily detected by shaking the sample with mercury, which will more or less blacken. Chloroform often contains alcohol. The usual test is agitation with alkaline permanganate, which should not turn green. The author found this test not to be reliable, as the greening may be caused by impurities in the potash. He therefore uses a solution of potassic permanganate in baryta-water. L. de K.

MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

"IMPERIALIN," A NEW ALKALOID. K. FRAGNER. *Berichte*, Dec. 12, 1888.—The author examined the bulbs of the poisonous crown imperial, *frittilaria* or *coronaria imperialis*, of the family *liliaceæ*, and prepared a new alkaloid, "imperialin," by two processes, of which the following is the best: The crushed bulbs are rubbed up with lime, and the mixture completely dried on the water-bath, and repeatedly extracted with hot chloroform, which is afterwards shaken up with water containing tartaric acid. The concentrated acid solution is precipitated with Na_2CO_3, and the precipitate freed from mother-liquor by means of the suction-pump. By repeated re-crystallisation from alcohol, the substance is obtained perfectly white. The yield is about 0·08 to 0·12 per cent. The formula Fragner calculates from his combustions (the carbon determinations vary considerably) is $C_{33}H_{60}NO_4$. The alkaloid gets yellow at 240°, brown at 248°, and melts completely at 254°. It crystallizes in short, colourless needles, is very slightly soluble in water, soluble in alcohol—especially so on warming—less soluble in ether, benzene, petroleum-ether and amyl-alcohol, dissolving very readily in chloroform. Its solutions have a bitter taste. Imperialin turns the plane of polarised light to the left. Fragner gives the specific rotation $[\alpha]_D = -35{\cdot}40°$. Tannin precipitates the solutions of imperialin salts yellow, flocculent; pottassium iodide, dark yellow, amorphous; potassio-mercuric iodide, reddish-yellow, flocculent; potassium bismuth iodide, orange-red; potassium cadmium iodide, white flocculent; potassium bichromate, yellow, crystalline; picric acid, yellow, flocculent. The following reactions of the dry substance are given. With concentrated H_2SO_4, a yellow colouration; mixed with sugar and then treated with H_2SO_4, the colouration is first yellowish-green, then pale-brown, flesh-coloured, cherry-red, and finally dirty-violet. Fröhde's reagent becomes greenish-yellow with it, Mandelin's, olive-green, reddish-brown, and finally dark brown. After trituration with cold H_2SO_4, KNO_3 and $KClO_3$ give orange-yellow colourations, which become dark reddish-yellow if the acid solution has previously been warmed. Warm HNO_3 colours the alkaloid yellow. With HCl a strong fluorescence is produced; on warming, a brownish-green colouration is obtained, which gradually turns brownish-red. No pressure is observed on opening tubes in which imperialin and HCl have been heated to high temperatures; therefore, no easily-removable methyl-groups are in the compound. Preliminary experiments seem to show that imperialin acts on the heart. A. L. G.

LAW NOTES.

ADULTERATED CHEESE.—At Glasgow Sheriff Court, on the 28th December, Sheriff Guthrie presiding, Alexander Moffat, 36, Orr Street, was charged, at the instance of Mr. Peter Fyfe, sanitary inspector, with having on Thursday, 27th September, sold 1 lb. of cheese which was not of the nature, substance, or quality demanded by the purchaser, in respect that it contained 14·12 per cent. of fat which was not butter fat. He pleaded not guilty, and was defended by Mr. T. C. Young and Mr. John A. Spens. Mr. R. G. Ross appeared for the prosecutor.

Mr. Spens objected to the relevancy of the complaint, on the ground that there was no averment that a little added fat took away from the character of the cheese.

The Sheriff repelled the objection on the merits, but said that the points might be raised as the case went on.

Dr. Tatlock, who analysed the cheese, reported that it contained 37·46 per cent. of casein, 12·56 of butter fat, 14·12 of foreign fat, 3·94 of mineral matter, and 31·92 of water.

Cross-examined by Mr. Spens: The proportion of butter fat in a skim-milk cheese ranges from 1 per cent. to 10 per cent. So far as he could judge, there was a greater proportion of butter fat in the cheese analysed than in the ordinary skim-milk cheese. A cheese made purely from sweet milk would contain about 33 per cent. of fat. That fat would be all butter fat. He detected the presence of foreign fat by the absence of butyric acid. That was the best method of testing. Butyric acid is always present in butter. There was, of course, some butyric acid in the cheese in question, but there was certainly not so much as there ought to be. He believed the foreign fat in the cheese was quite wholesome, and he believed the cheese was quite wholesome and nutritious. Personally, he would as soon eat these cheeses as skim-milk cheese. There was more fat in them than in skim-milk cheese, but less of the other nourishing ingredients. The foreign fat could not be got in without taking out some of the other nourishing ingredients, such as casein. The percentage of casein in skim-milk cheese ranges from 35 to 45 per cent. In this particular case he found 37·46 per cent. of casein. The percentage of water should be 25 to 35 per cent., and of mineral matter from 3 to 6 per cent. Nourishment is got from the fat, but he would not call it the principal source of nourishment, for there is also casein. There was cheese made in America mixed with fat. It was sent here in large quantities, and sold as cheese, but he did not allow that it was cheese.

By Mr. Ross: He believed the latter class of cheese was called margarine cheese in America. Skim-milk cheeses made in this country are very dry. The addition of foreign fat gave the cheese an appearance of being richer—more like a sweet-milk cheese.

This closed the case for the prosecution.

Preceptor Alexander Osborne was the first witness called for the defence. He stated that he is a partner of the firm of Alexander Osborne and Sons, cheese merchants, Candleriggs, and a shareholder in the Creamery Company, Dunragit, who made the cheese in question. What was formerly known as skim-milk cheese is almost an unsaleable article now. It is of very poor quality, and to make a saleable article they found it necessary to improve it by adding a little fat, which made it rather dearer than skim-milk cheese. This is about the cheapest quality of cheese sold in the market. The retail price of the lowest quality of sweet-milk cheese at present ranges from 6d. to 10d. Sweet-milk cheeses are commonly understood to be made from milk that is not skimmed, but cheeses that are made from milk as it comes from the cow are properly called whole-milk cheeses. No one could expect to get whole-milk cheeses at 4d. per lb. The price indicated that they were something different from what were sold as sweet-milk cheese. The cheeses in question were superior to skim-milk cheeses, because fat was added that made them soft and more digestible. The added fat compensated for the cream that was taken away, and made the cheese very nearly, if not quite, as good as the dearer article sold as sweet-milk cheese, and much better than some of them. Originally all Scotch cheeses were skim-milk cheeses, but when the system was changed in the parish of Dunlop the cheeses made there from whole milk were called Dunlop cheeses, to distinguish them from what were called Scotch cheeses.

By Mr. Ross: No person tasting the cheeses in question would distinguish them from ordinary cheeses, except that they might be rather better. Dunragit cheeses have been in the market for two or three years, and the company would prefer that shopkeepers should sell them by that name. Cheeses mixed with foreign fat have been made elsewhere for a long time, but not in Scotland, so far as he was aware.

Mr. Andrew Clement gave similar evidence. The fat introduced into the cheese is cow fat, pure and wholesome.

The Sheriff remarked that if there had been an attempt to sell the cheese at 6d. the case would have been different, but it was sold at 4d., which was not a price above its value.

Mr. Ross contended that when a purchaser asked for cheese he was entitled to get cheese that was made entirely from milk.

The Sheriff said he was satisfied that the case fell under the proviso in the 6th section of the Food and Drugs Act, "Where any matter or ingredient not injurious to health has been added to a food or drug because the same is required for the production or preparation thereof as an article of commerce in a state fit for carriage or consumption." That must be read as meaning that it was intended to prepare the article for consumption with reference to the price that was to be paid for it and the purpose for which it was intended. It was quite clear that there was not in this case anything done "fraudulently to increase the bulk, weight, or measure of the food or drug, or conceal the inferior quality thereof." What the result of another prosecution for a similar case might be it would be wrong for him to prognosticate, but from the evidence it was clear that this case did not fall within the section. The case was accordingly dismissed.

In his report, which was included in the minutes of the Health Committee, Mr. Peter Fyfe, sanitary inspector, in referring to the above case, says that, in giving decision in favour of the accused, his lordship said that the sixth section of the Adulteration of Food and Drugs Acts must be read as meaning "that it was intended to prepare the article for consumption with reference to the price that was to be paid for it and the purpose for which it was intended." It was quite clear that there was not in this case anything done "fraudulently to increase the bulk, weight, or measure of the food or drug, or conceal the inferior quality thereof." The principle here laid down that "foreign" fat may be introduced legally into cheese, so long as the fat used is not injurious, and the cheese is sold at a moderate price, is (says Mr. Fyfe) a new one, which places cheese in the list of *manufactured* articles, and displaces it as a purely milk product. This lays the market open to large importations of "imitation cheese" from America.

Mr. Crawford said he did not desire to say very much about the case. The Committee were not satisfied with the decision, and they had the matter fully under view, and would in course of time take such measures as seemed to them necessary in the circumstances.

APPOINTMENTS.

Dr. H. Leffmann has been appointed Pathological Chemist to the Jefferson Medical College Hospital, Philadelphia.

Mr. Beam has been appointed Demonstrator of Chemistry at the Pennsylvania College of Surgery.

THE ANALYST.

MARCH, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

AN ordinary meeting was held at Burlington House, on Wednesday, the 13th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

The adjourned discussion on Dr. Dupré's paper on "A Recent Case of Alleged Injurious Pollution of the River Medway by the Sewage of Maidstone," was then resumed and concluded.

The following papers were subsequently read and discussed :—

"On the Composition of Milk and Milk Products." By Dr. Vieth.

"An Improved Method for the Analysis of Fats." By Dr. J. Muter and L. de Koningh.

"Note on Phytosterin." By Otto Hehner.

"On Density." By H. Droop Richmond.

The next meeting of the Society will be held at Burlington House, on Wednesday, the 13th inst., at 8 o'clock.

PEPPER ANALYSIS AND THE OCCURRENCE OF PIPERIDINE THEREIN.

BY WILLIAM JOHNSTONE, PH.D., F.I.C., F.C.S., ETC.

(*Read at Meeting, January,* 1889.)

MR. PRESIDENT AND GENTLEMEN,

The subject of the paper I have the honour of reading before you this evening is "Pepper Analysis and the Occurrence of Piperidine therein."

What is known as black pepper is the dried immatured fruit of *Piper Nigrum*, belonging to the *Natural Order Piperaceæ*.

The shrub or plant is a native of tropical regions, and is cultivated in various parts of the Indian Archipelago and in the West Indies, and that imported into this country comes principally from the Islands of Malacca, Java, Borneo, and Sumatra.

The plants of the order of Piperaceæ contain an acid resin, a volatile oil, a volatile alkaloid, and a crystalline substance called piperine. They possess pungent aromatic, astringent, and narcotic properties.

Botany. Perennial, *Stem.*—Its stem is 8 to 12 ft. long, trailing or climbing, shrubby, flexuose, and dichotomously branched, jointed, swelling at the joints and often throwing out radicles there, which adhere to bodies like the roots of ivy, or become roots, striking into the ground.

Leaves.—From 4 to 5 in. long, alternate, distichous, broadly ovate, acuminated, of a full green and glossy colour, paler beneath, five to seven-nerved, the nerves connected by lesser transverse ones or veins, and prominent beneath.

Inflorescence.—Spikes opposite the leaves, chiefly near the upper ends of the branches; shortly stalked, pendulous, 3 to 6 in. long; petioles rounded, from ½ to nearly 1 in. long.

Flowers unisexual or hermaphrodite, small, whitish.

Fruit.—Distinct, braccate, ripening irregularly all the year round, about the size of a pea, at first green, then red, afterwards black, covered with pulp.

The black pepper of commerce consists of the entire berries, with the pulp adhering, gathered before they are quite ripe and dried in the sun. The commercial varieties are at least ten in number, viz., Acheen, Alleppy, Kampoot, Lampong, Penang, Siam, Singapore, Tellicherry, Trang, and Long pepper, the names indicating the localities from whence they are obtained.

Blyth remarks that "the differences which these different varieties of pepper present *to the eye* are evident enough when the several samples are at hand for comparison," a statement I by no means agree with, "but it takes a very practised observer to identify a solitary sample; and if samples of each of the kinds were mixed together, it is doubtful whether an adept even could separate the berries again, identifying each sort with any correctness." Very doubtful, I should say, indeed.

The merchant, however, relies more upon the weight than the appearance. Taking a handful of peppercorns, he can tell in a moment whether it is a light or a heavy sample, and thereby judges the quality of the pepper.

That there is a difference in the weight of the various varieties of the different peppercorns is evident from the results obtained by the author, who carefully three times weighed 100 berries of each kind, which gave the following average results:—

Average of Three Weighings of 100 Peppercorns.

Acheen	5·1976 grms.
Alleppy	3·8438 "
Kampoot	4·4540 "
Lampong	3·5410 "
Penang	3·9028 "
Siam	4·2776 "
Singapore	4·5338 "
Tellicherry	4·4421 "
Trang	4·8101 "
Penang, white	4·9360 "
Siam, "	5·1441 "
Singapore "	4·6936 "

The pepper of commerce, whole or ground, is therefore a mixture of the different kinds of peppers, and is a condiment which has had a good deal of attention paid it lately, owing to some successful prosecutions which took place at Liverpool some time ago, when a large quantity of ground pepper was condemned as being adulterated with a material known by the name of *Poiverette*, a hard, tasteless, woody substance, absolutely worthless as a condiment, imported expressly into this country from Italy, for no other purpose than of fraudulently increasing the weight of ground pepper.

It was not, however, wholly owing to the addition of *Poiverette* to pepper that I was induced to undertake the following analysis, although it tended greatly in that direction, but to the great divergency of opinion that seemed to exist amongst various authorities as to the amount of piperine contained in pepper.

The standard works of reference are also at variance with one another, Watts' Dictionary stating that Penang white pepper gave a mean of 5·24 per cent. and Singapore white 9·15 per cent., whilst Singapore black gave a mean of 7·15 per cent.; Sumatra 8·10 per cent. Mr. Heich (ANALYST, vol. xi., p. 188), from 5 to 9 per cent., Mr. Winter Blyth, from 1·8 to 5·5 per cent., Dr. Muter in his "Organic Materia Medica" (third edition), from 2 to 3 per cent., and Ure's Dictionary (sixth edition) a fraction of a grain, and lastly, Dr. Stevenson, in the ANALYST, vol. xii., p. 144, from 6·62 to 7·14 for black pepper, and 6·47 per cent. for white pepper.

Owing, therefore, to such conflicting evidence, with considerable variation in the amount of piperine said to be contained in similar peppers, I determined to investigate the subject myself.

I have done so, and the results obtained in that investigation furnish me with the contents of this paper, and although I have not been able to reduce the limits of variation (but on the other hand to increase them), I have been rewarded by the discovery of the fact that pepper contains a volatile alkaloid, and I have every reason to believe that it is piperidine, from the results I have obtained upon the analysis of its platinum salt:—

		Found.	Calculated.
C	120·0	20·02	20·60
H	24·0	4·32	4·15
N	28·0	5·01	4·80
Pt	197·4	33·93	33·89
Cl	213·0	36·62	36·56
$2(C_5H_{11}N.HCl)PtCl_4$ ..	582·4	99·90	100·00

In entering upon this investigation I had in the first place to obtain genuine specimens of pepper, and through the kindness of my friend Mr. Worcester, I obtained from Messrs. Lewis and Peat, well-known brokers in the City, thirteen specimens, and I now take this opportunity of thanking them for their liberality and the trouble they took to obtain them for me.

The samples as received by me were taken from the bulk, that is, dock or cargo samples, and are therefore undoubtedly genuine, and are representative samples of the pepper which is sent to this country, having undergone no particular process of cleaning other than having simply been screened by means of a sieve, containing seven meshes to the inch, before being ground.

Having obtained such magnificent samples, my next consideration was the adoption of a process for the estimation of the piperine, which would give constant and accurate results, as I had grave doubts as to the accuracy of the alcohol method usually followed, that is, extracting with alcohol and then purifying by potash, and subsequently again crystallising from alcohol.

At this point I therefore directed my attention to the old but well-known fact, that piperine, when treated with caustic potash and alcohol, splits up into piperidine and pipric acid, as represented by the following equation :—

$$C_{17}H_{19}NO_3 + KHO = KC_{12}H_9O_4 + C_5H_{11}N$$
$$C_{17}H_{19}NO_3 + H_2O = C_{12}H_{10}O_4 + C_5H_{11}N$$

Piperidine, as you are all well aware, is a volatile alkaloid possessing strong alkaline properties (B.P. 106° C.), so the idea immediately occurred to me that if the above equation could be made to work in practice so as to yield the theoretical quantity of piperidine, the difficulty would be solved, there being no difficulty experienced in distilling off the piperidine and titurating the distillate with standard acid.

The next thing, therefore, was to prove the above reaction, so having some very fine crystals of piperine in my possession, which I had obtained from Messrs. Macfarlan and Co., of Edinburgh, some years ago, and which I had every reason to believe were pure, but erring on the safe side I re-crystallised them, and subjected various quantities to the action of caustic potash and alcohol.

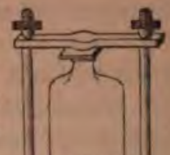
A series of misfortunes and unsatisfactory results followed, so unsatisfactory that I got quite disheartened, concluded that it was a miserable failure, and allowed the subject to remain at rest for over eighteen months, when fortunately one idle day I again returned to the subject with increased vigour.

The trouble I had to encounter before was the difficulty of keeping the stoppers of the bottles tight when subjected to heat and pressure, as the digestion was carried on in the water-bath at a temperature of 100° C.

If one was fortunate enough to get them to remain tight under pressure it was 100 to 1 that when allowed to cool, and you attempted to remove the stopper, it would be found hard and fast, no alternative being left but to knock off the neck; otherwise, if it was got out without breaking the bottle, I always found a great loss.

After a dozen bottles had been used up in this manner, not counting the ones that burst, I found that in three instances I had obtained practically theoretical results (which will be seen by referring to the three first estimations in the accompanying table), so encouraged by my results, and at the same time determined to overcome the difficulty, if possible, and make the process a working success, I designed the following little arrangement, with which I have obtained actually theoretical results, which I have tabulated in the diagram before you, the first three estimations, as already stated, having been made in an ordinary stoppered bottle.

	PIPERINE.	
Taken.	Found.	Per Cent.
·8400	·83075	98·89
·2520	·24937	98·95
·5900	·58450	99·06
·4320	·43170	99·93
·7640	·76380	99·97
·5288	·52212	99·85
·3530	·35197	99·70
1·0108	1·01052	99·97



This instrument, as you will observe, consists merely of an ordinary 4-oz. bottle with its mouth ground flat, and is simply closed by placing a circular piece of sheet india-rubber over it, and then placing it in its holder, putting the clamp in position, and finally making it perfectly tight by screwing down the two screws made for the purpose, put into the water-oven, digested at a temperature of 100° C. for from four to six hours, when the reaction is complete, then removed from the oven and allowed to cool.

When cool it will be found that the bottle has remained perfectly tight, none of the contents having escaped, so all that now remains to be done is to unscrew the clamp, and transfer the contents of digestion bottle to a distilling flask fitted with a condenser, and distil the piperidine and titurate the distillate with standard acid.

Having, therefore, satisfied myself of the accuracy and adaptability of the process, the next thought occurred to me to try the estimation of the piperine contained in the pepper direct, that is, digesting the ground pepper with caustic potash and alcohol for six hours, and then distilling as before.

Ten grms. of ground pepper was carefully transferred to the digestion bottle, 30 c.c. of alcohol added, and then about 3 grms. of caustic potash dissolved in 25 c.c. of water also added, the indiarubber placed in position and the clamp screwed down, and then digested for six hours, allowed to cool, opened at convenience, and contents of the bottle washed into large flask with plenty of water, distillation commenced and carried on until the distillate coming over is neutral, aliquot portions of which are then titurated with $10 \frac{N}{H_2SO_4}$ using methyl orange as an indicator.

Several experiments were performed in this manner on one sample of pepper, concordant results being obtained in every instance, which established the reliability of the process; consequently all the samples of pepper were treated in this manner, and the results so obtained are tabulated in the table before you.

The piperine estimations having proved successful, I decided to make a complete analysis of the various samples, the results of which I have the pleasure of laying before you this evening.

I will now, therefore, describe in detail the processes employed by me so as to obtain the figures I have placed upon the diagram.

Moisture.—The moisture is estimated by the ordinary method of drying a weighed portion of the ground pepper in a platinum capsule at 100° C. in the water-oven until the weight remains constant, and then noting the loss in weight.

Oil.—The volatile oil is estimated by distilling 20 grms. of the ground pepper with water in a large flask, shaking the distillate several times with ether, and evaporating the separated ether in a weighed flask at a very low temperature, and finally drying over sulphuric acid.

Piperidine.—Twenty grms. of the ground pepper are distilled with water in a similar manner as that for the oil estimation until the distillate is neutral (450 c.c. being about the quantity necessary to distil). An aliquot portion is then titurated with $10 \frac{N}{H_2SO_4}$ using methyl orange as an indicator. In the piperidine estimation it occurred to me that the process of distilling with water might be done away with, and the pepper put into a boat and then into a hot-air tube, heated to 230 to 240° F., a constant current of air being aspirated through it all the while, the air ultimately passing through a bulb of standard acid.

Working in this manner, and after an hour's treatment, the acid was washed out of the bulb and its equivalent of soda added, the excess of alkali being titurated back with $10\frac{N}{H_2SO_4}$ using methyl orange, as usual, as indicator. The results obtained by working in this manner are interesting on account of only a third of the piperidine being obtained in this way, or rather only a third of that obtained when the pepper is distilled with water.

The following are the results obtained in duplicate when working in the manner just described, using 5 grms. of the pepper:—

No. Sample	I.	II.	III.	IV.	V.	VI.	VII.
	0·13	0·106	0 16	0·25	0·11	0·19	0·19
	0·13	0·105	0·12	0·25	0·10	0·20	0·17

No. Sample	VIII.	IX.	X.	XI.	XII.	XIII.	XIV.
	0·29	0·17	0·127	0 093	0·076	0·085	0·272
	0·25	0·17	0·127	0·093	0·076	0·085	0·271

These results tend to suggest that the piperine was to some extent hydrolysed by the action of prolonged boiling with water, but such is not the case for if pure piperine is boiled with water no piperidine is obtained, and it is also disproved by the fact that there is no difficulty in getting the distillate neutral; the amount of piperidine distilled represents a mere fraction of the piperine remaining in the pepper.

I am rather disposed to the theory that there may be a particular ferment contained in the pepper capable of producing the hydrolisis of piperine when boiled with water, or it may be due to the existence of another alkaloid more easily hydrolysed than piperine, anyhow these are the results obtained, and any one is welcome to continue the investigation further if they should feel so disposed

Ash.—The dried sample used for the moisture estimation is incinerated at a very low temperature, the most convenient arrangement for this work being a jar-muffle, which is infinitely superior to any kind of burner. The residue obtained is the mineral or inorganic matter, and this in turn is then treated with water and then with dilute acid, so as to obtain the portions soluble and insoluble in those menstruums, the insoluble in acid portion, representing the proportion of sand present, and is of special importance.

Piperine.—Ten grms. of ground pepper are digested in a closed bottle similar to that already described, from four to six hours, with 3 grms. of caustic potash dissolved in 25 c.c. of water and 25 c.c. of alcohol. When the digestion is finished, the apparatus is allowed to cool and its contents then carefully washed into a large flask, a few pieces of tobacco-pipe stalks being also put into the flask so as to prevent the violent bumping which would otherwise occur, distillation commenced and continued until the distillate coming over no longer shows signs of alkaline reaction, which is generally the case when about 750 c.c. have passed over. The distillate is then made up to one litre, and titurated as before with $10\frac{N}{H_2SO_4}$ using methyl orange as indicator.

Fibre crude.—Between 3 to 5 grms. of the ground pepper are placed in a flask and covered with 50 c.c. of dilute sulphuric acid (5 per cent. or 50 grms. of sulphuric acid per litre), to this 150 c.c. of water are added and the liquid boiled for half an hour, the flask having previously been attached to an upright condenser.

After subsidence, the supernant liquid is run off by means of a syphon into a beaker, and the residue is then twice boiled for half an hour with 200 c.c. of water. After removing as much of the clear liquid as possible, by decantation, the residue, to which is added any sediment that may have become deposited in the beaker containing the acid extract, is next boiled in the same manner with 50 c.c. of dilute caustic potash (50 grms. of KHO in 1,000 c.c.) and 150 c.c. of water for half an hour, and after subsidence the supernant clear liquid is syphoned off into another beaker, and the residue is again extracted by boiling twice with 200 c.c. of water.

The residue obtained after the above process of extraction has been carried out, together with any sediment deposited in the beaker containing the alkaline extract, is now thrown upon a weighed filter and washed well with boiling water, alcohol and ether. The filter and residue are then dried at a temperature of 105 to 110° C. and weighed, and then, in order to ascertain the small proportion of ash which the woody fibre contains, also the accompanying sand, the filter is ignited and the ash found deducted from the previous weight.

This is merely an approximate determination, as the term *crude fibre* designates nothing beyond the fact that it does not consist of pure cellulose, but that a certain amount of ligenous substances are insoluble in acid and alkaline solutions of definite strength, and alcohol and ether, after definite treatment, such as already described, furnishing results which are of some comparative value, but still leaves much to be desired.

Nitrogen and Albuminoids.—The nitrogen was determined in the usual manner with soda-lime.

Alcoholic Extract.—Two grms. of the ground pepper are extracted for 24 hours with 95 per cent. alcohol.

The pepper under examination is placed in a tube, which in turn is inserted in a Soxhlet's extraction apparatus.

The tubes used for this purpose are ordinary test tubes cut in two, the upper half having a piece of fine linen (previously washed and extracted) tied over the mouth end of the tube, then a wad of extracted cotton-wool of sufficient thickness is placed inside the tube, upon the linen so as to prevent any of the solid particles of the sample finding their way into the receiving flask, and then another wad of cotton-wool is packed on the top of the sample and the whole inserted in the Soxhlet apparatus, and the extraction continued, as already stated, for 24 hours. The alcohol is then distilled off, and the flask dried in the water-oven until weight remains constant.

I must here remark that it is necessary to distil the recovered alcohol with dilute sulphuric acid if it is to be again used for any other purpose, so as to get rid of the piperidine which it now contains.

Starch.—Before proceeding to convert the starch into dextrine, the pepper must be carefully extracted with 95 per cent. alcohol, so as to remove substances other than starch which are acted upon by the acid, and also which have the power of reducing Fehling's solution, such as piperine and piperidine, both substances being possessed of the power of reducing Fehling's solution.

The residue contained in the Soxhlet tube is, therefore, readily available for this purpose, having been thoroughly exhausted with alcohol.

Without drying, therefore, it is carefully washed off the cotton-wool into a suitable flask, with about 200 c.c. water and 20 c.c. HCl (sp. gr. 1·121) added, the flask connected with an upright condenser and heated in the water-bath for 3 hours.

After cooling, the liquid is filtered, and the filtrate very carefully neutralised with caustic soda and diluted, so as to measure 500 c.c.; finally titurated with Fehling's solution, and the results calculated to starch or any other desired basis.

A few drops of chloride of zinc may be added, so as to assist clarification of the liquid before filtering.

Extraction with alcohol is indispensable, without which the results are most uncertain and unreliable. Proper and careful extraction with uninterrupted boiling are the two most important conditions to be attended to.

These results, therefore, which I have this evening placed before you, were obtained by the methods I have just described, and although individually I am disappointed with them, I must admit, nevertheless, they are of considerable value, as thay are representative analyses of several varieties of pepper, obtained from various quarters of the globe, and as met with in commerce and as shipped to this country.

The moisture and oil determinations are of no practical value, and the same may be said of the piperidine and piperine; the piperidine being in too small quantity to be of any use, the piperine, on the other hand, exhibiting large variations, showing a maximum of 13·03 per cent., with a minimum of 5·21 per cent., and giving a mean of 8·25 per cent.

The albuminoids and matter soluble in alcohol are also of little practical value (except that an excessive amount would reveal the presence of some foreign seed rich in nitrogen), so that we are left with the starch, fibre, and ash as the only data which approaches to anything like reliable, contant, or uniform amount. Fortunately in two of these we have very reliable data, viz., in the ash and fibre. Too great a value, in my opinion, has been placed upon the amount of starch, as will be seen in referring once more to the diagram, a maximum of 42·45 per cent. being obtained, with a minimum of 29·60 per cent., giving a mean of 36·02 per cent. for the black peppers.

It may be of more value in judging white peppers, but as I had only three samples of white, my remarks do not, therefore, apply to white pepper.

An increase of fibre indicates the addition of a more fibrous material than pepper. The fibre will be observed to reach a maximum of 15 per cent., with a minimum of 10 per cent., and a mean of 12 per cent., so that we can easily accept 20 per cent. as a fair basis for calculating the amount of fibrous material.

When we come to the consideration of the ash, however, we have very reliable data, the amount of ash immediately indicating the addition of any material containing a larger proportion of inorganic matter than pepper.

The ash is undoubtedly a most reliable and valuable constituent, and it will be observed that not in a single instance has it reached 5 per cent. If we leave out the Long pepper, we obtain a maximum of 4·65 per cent., with a minimum of 3·51 per cent., and which gives a mean of 3·96 per cent. Anything, therefore, above 5 per cent. must be looked upon with suspicion.

Another valuable indication is the solubility of the ash in water and in hydrochloric acid.

In water we have a maximum solubility of 2·72 per cent., a minimum of 1·41 per cent., with a mean of 1·93; and the insolubility in hydrochloric acid gives a maximum of ·62 per cent., with a minimum of 0·06 per cent., and a mean of 0·82 per cent. (Long pepper gives 1·47), so that anything over 1 per cent. of insoluble matter in hydrochloric acid ought to be reported against as containing sand or dirt.

	Moisture.	Oil.	$C_5H_{11}N$	Ash.	$C_{17}H_{19}NO_3$	Fibre.	Albuminoids.	Starch.	Soluble in C_2H_6O.	Undetermined.	Total.		Solubility of Ash in		
												N.	H_2O.	HCl.	SiO_2
Acheen ..	15·15	1·51	0·50	3·79	12·21	10·00	2·34	42 45	0·84	11·21	100·	1·06	1·41	1·74	0·62
Alleppey ..	15·36	1·87	0 44	4·66	13·03	13·10	2·62	29·60	2·12	17·20	100·	1·34	2·72	1·87	0·06
Kampoot ..	13·82	1·63	0 55	3·51	8·13	11 65	5·37	37·50	9·57	8·27	100·	1·42	1·50	1·70	0·31
Lampong ..	15·22	1·42	0 77	4·46	11·05	15·05	7·25	30·80	4·55	9·43	100·	1·82	2·15	2·10	0·21
Long Pepper ..	12·26	1·56	0·34	7·57	7·15	13·75	6·93	32·10	3·50	14·84	100·	1·51	2·18	3·92	1·47
Penang, W.C. ..	15·04	0·98	0·46	4·14	6·41	11·70	8·37	41·65	5·44	5·81	100·	1·72	1·71	1·63	0·56
Siam	14·06	1·29	0·76	3·65	6·89	12·25	6 93	40·15	6·26	7·76	100·	1·57	1·64	1·67	0·34
Singapore ..	14·72	0·99	0·72	3·64	5·72	10·75	6 31	32·18	5·55	19·42	100·	1·40	2·00	1·48	0·16
Tellicherry ..	14·24	1·01	0·39	3·87	8·25	12·15	5 87	33·33	2·60	18 29	100·	1·40	2·26	1·55	0·06
Trang	14·02	1·40	0·47	3·96	5 21	10·70	6·68	37·50	4·54	15·52	100·	1·49	1·74	1·92	0·30
Penang, W. ..	14·94	0·53	0·34	2·47	8·87	4·35	2·62	51·00	0·46	14·42	100·	0·90	0·31	1·97	0·19
Siam, W. ..	13·13	1·41	0·21	2·22	7·79	4·45	6 00	53·50	0·11	11·18	100·	1·37	1·18	0·95	0·09
Singapore, W. ..	15·62	1·14	0·42	1·07	8·66	4·20	7·00	52·00	1 76	8·13	100·	1·61	0·30	0·62	0·15
Husks, etc. ..	12·54	1·74	0·74	16·34	6·32	22·88	6·50	11·80	4·23	16·91	100·	1·47	1·10	6·71	8·53

THE WESTPHAL BALANCE.

BY W. F. K. STOCK, F.C.S., F.I.C.

(*Read at Meeting, January*, 1889.)

IN Mr. A. H. Allen's most valuable and painstaking paper, "On Some Abnormal Samples of Butter" (ANALYST, January, 1889), there are some remarks on the Westphal balance which will come as a positive shock to any chemist who may have been too confiding in the performance of an untried instrument.

Mr. Allen did not mention what his balance had cost, but it may safely be said that the cheaper such an instrument is, the more likely will it be to possess defects, for the reason that the actual value of the material used in its construction is as nothing to the labour-cost of its truthful graduation and the accurate adjustment of its weights.

The ease and rapidity with which the densities of liquids can be ascertained at any temperature between 0° and 99·5° C. by the use of the Westphal balance give it a place in the laboratory which nothing else could well supply, and I should very much regret if analysts, and especially the members of this Association, should find it necessary to give the cold shoulder to this handy little machine, for which reason I should like to say a word in its favour. I cannot do this better than by giving my own experience of it. The balance I use was supplied by Oertling, at a cost of three guineas. It has a set of "rider" weights in duplicate. Before taking it into work, I made a rigid inquiry into its performance, thus: The graduations on the beam were measured up by means of a fine springbow divider worked with a screw; the spaces were taken off with a scale of equal parts (by Archbutt) and found to be accurate. The displacement of the plummet in distilled water at 15·5° C. was got on a short-beam balance by Bunge, and the whole of the rider weights were weighed on the same balance, with the beam oscillating, by which means a degree of accuracy beyond the fourth place of decimals was reached.

The following table shows the variations in the riders:—

Number of Weight.	Weight in Grammes in Air.	Relation to Unity. Plummet displacement = 6·48 grms.
1	6·4806	1·00007
1*a*	6·4796	·99992
2	·6482	·10003
2*a*	·6483	·10004
3	·0655	·01010
3*a*	·0655	·01010
4	·0070	·00108
4*a*	·0071	·00109

Density of rectified spirit by Westphal .. .. ·8383
" " " Bottle .. .. ·8385

A glance at the third column of the table will show that the maximum deviation from unity is 1 in the fourth place of decimals, a result which is creditable to the maker, since the balance was supplied in the ordinary way, without any attempt at selection.

The simple law which governs the action of all balances of the Steelyard type tends to aggravate error in direct proportion to the increase of mass in the weight, and the use of rider weights for accurate work on chemical balances lies within very narrow limits, nothing beyond a centigramme being common. In the Westphal balance, however, the whole displacement, which may amount to 10 grms. or more, is called unity, and therefore, unless very palpable imperfections are present, they do not practically vitiate the results; and seeing what can be had at a moderate cost, as in the case of my own purchase, I think there can be no doubt that if another guinea were added to the price, and the extra cost were devoted entirely to the correction of spacing and adjustment of weights, a reliable and trustworthy instrument would be the outcome. The maker could then afford to issue a guarantee with each instrument.

DISCUSSION.

Mr. ALLEN said the instrument which he had referred to in his paper on "Some Abnormal Samples of Butter" was a Westphal balance, obtained from Messrs. Becker and Co., of Maiden Lane, and, he believed, cost 28s. It was of the ordinary kind sold by that firm. The fault was simply in the inaccurate division of the beam at the eighth point. In other respects it was a perfectly satisfactory instrument, and he had used it for a considerable time with every satisfaction. It must be remembered also that the gravities commencing with the figure 8 were all comparable among themselves, and it was only when it was desired to compare densities obtained by that instrument with those determined by the specific gravity bottle that any discrepancy became important. Still, he had thought it his duty to mention the matter, as he felt that his brother analysts were very probably recording figures in which the same error was involved. He had recently obtained a highly satisfactory Westphal balance from Sartorius, at £2 10s., and also one from Oertling, at £3 3s. Both of these were admirable instruments, and in the case of Oertling's instrument the bearings were of agate, but in both balances the knife edges were of steel. He rather preferred Oertling's balance, as it was intended for use with a 5 c.c. plummet instead of a 10 c.c. plummet, which latter was supplied with the Sartorius instrument.

(Conclusion of Society's Proceedings.)

ABNORMAL BUTTERS FROM IRELAND, DENMARK, AND SWEDEN.

By CHARLES ESTCOURT, F.I.C., F.C.S.

IN April, 1885, a client of mine delivered a sample of Irish butter, duly sealed, and labelled as having been purchased under the Food and Drugs Act.

The Public Analyst for the Borough where the sample was purchased, had condemned it as being a mixture of foreign fat, and my analysis confirmed this result.

SO-CALLED IRISH BUTTER.

Specific Gravity at 210° F.	Insoluble Fatty Acid.
865·1	92·07

The Analyst for the Borough had condemned this upon the result by Reichart. Upon investigating the matter I found that my client had purchased this butter from a very large firm in Ireland, who were, he said above suspicion, one of the firm, indeed, was a member of Parliament.

Certificates for the butter in question had been given by two of the first chemists in Dublin, and it was said even to have passed Somerset House. When the case came before the magistrates however, the Irish firm did not, and my client, acting under my advice would not, ask the magistrates to send the sample to Somerset House.

With the reputation for honesty which all the parties concerned bore, it only needed a possibility of seeing the cows milked, and a supervision of such milking by *inexperienced* persons to prove this "an abnormal sample of butter."

Fortunately, however, under pressure from my client, we obtained the whole secret of the process of manufacture.

Last year's butter was purchased and washed free from all rancidity, churned again with skim milk, oleomargarine, and about 10 per cent. of nut oil, the result being a perfect butter so far as flavour was concerned.

In 1884, two samples of Danish butter were submitted to me for analysis, from one of the boroughs for which I am analyst. I was of opinion that they were adulterated, but before any proceedings were taken, duplicates of the samples were sent to Somerset House, with the result that they pronounced them pure. The following are the analyses:—

	Somerset House.		Estcourt.	
	No. 1.	No. 2.	No. 1.	No. 2.
Insoluble Fatty Acid	89·35	88·85	89·45	88·59
Soluble Fatty Acid	4·18	4·24	4·20	4·01
Specific Gravity at 210°F.	..	..	865·70	866 0

These results would, I am convinced, be now held by all analysts, including Dr. Bell himself, sufficient proof of adulteration. The soluble fatty acids being lower than those even recently obtained by Mr. Allen from his abnormal butters.

It will be remembered that this was before the period when analysts believed that a mixture containing any considerable quantity of butter-fat would pay. It had always been supposed that adding a flavouring of butter-fat to foreign fat was the ordinary method of manufacturing butterine.

In 1885, some clients of mine who are very large purchasers of butter, and have special purchasing agencies in both Denmark and Ireland, and who, I may say, are the largest retailers of butter in Great Britain, brought me a sample of a consignment purchased by their agent in Denmark. The following were the results of the analysis:—

	Dr. Bell.	Mr. Allen.	Estcourt.
Specific Gravity at 100° F.	908·42	..	..
Specific Gravity at 210° F.	..	..	862·50
Insoluble Fatty Acids	90·08	..	90·12
Soluble Fatty Acids	3·48	..	3·50
Reichart 2½ grammes	..	8 9	..

I pronounced this to be a mixture, and both Dr. Bell and Mr. Allen agreed with me.

On reporting the results to my clients, they informed me they had submitted the sample for analysis solely because their buyer had written that "this shipment was poor in quality."

In the face even of these analyses, the producers asserted that the sample was genuine.

In August, 1888, a sample of Swedish butter was received from the butter importer in this country, by the hands of a solicitor at Wigan.

This sample was duly sealed, labelled, and numbered 402 by the Inspector of the Corporation of Wigan.

The analyst for Wigan found that the sample was a mixture, and, on analysis, I agreed with him. When the case came into court, the butter importer asked for the sample to be sent to Somerset House, and this was accordingly done.

On analysis by Dr. Bell and his colleagues, they agreed with us in pronouncing the sample a mixture.

The following are the results of analysis :—

	Somerset House.	Estcourt.	
Specific gravity at 100° F. ..	909·4	..	Wigan analyst has been requested to give results but has not done so.
" " 212° F. ..	..	865·5	
Insoluble fatty acid ..	90·0	89·8	
Soluble " ..	3·32	3·40	

Shortly afterwards two more samples, G, N, were delivered to me by the same butter importer, and I found on analysis the following results :—

	G.	N.
Specific gravity at 212°	864·5	864·0
Insoluble fatty acid	89·7	89·0
Soluble "	3·53	3·57

These were reported to be from the same farm as the Wigan 402, and had been analysed by Professor Stein, with the same result as 402.

Later on, the gentleman who represents the Danish agricultural interests in this country, in company with the butter importer, called upon me. I explained to them my view as to the incompleteness of the test of supervision applied at the Swedish farm, whence G and N were taken, and on a suggestion of the agent that it might be essential that I myself should visit the farms, I expressed my willingness to place several days at their disposal.

Having personally supervised the milking of many hundreds of cows of various dairies, I may, without egotism, say that my experience is unique as to the possibilities of fraud or error committed during such operations. Mr. Carter Bell, the analyst for Salford, and two of our Manchester food inspectors, are the only persons having to my knowledge a similar experience.

Shortly after this interview, the gentleman representing the Danish agricultural interests wrote to the Manchester papers explaining how carefully the supervision of the milking had been carried on, from which Professor Stein had obtained his samples G and N.

In reply to this I pointed out that the supervision extended only to the churning of milk, which itself came from no one knew where.

I also stated that no genuine sample of butter from England, Scotland, Wales, and Ireland had yet been analysed giving such abnormal results.

As it seemed incredible that Denmark or Sweden should possess a special breed of cows, or grow naturally special food, in my reply I placed my services for a few days at the disposal of the gentlemen who interviewed me, in hopes that this extraordinary mystery might be unravelled.

From that time until the 3rd of January, 1889, I heard no more of this matter; then, to my surprise, I saw in the ANALYST the paper on abnormal butters.

From this paper, and the analysis of samples B and O, Danish butter, it appears that we are expected to infer that the samples of Swedish butter, Nos. 402 and G and N, were similar in character to B and O. The analyses negative this assumption at once While no one would hesitate to pronounce Nos. 402 and G and N adulterated, very few analysts would venture to assert that O and B might not be *abnormal* butters.

The figure for soluble fatty acids is about equal to the lowest figure to be found in Dr. Bell's book, and the specific gravities approach exceedingly near the lowest normal. The visit to Denmark has therefore not materially advanced our knowledge of the butter question. Whatever may be the opinion of analysts with regard to the proof of genuineness of B and O afforded by Mr. Allen's paper, no one can doubt the lack of such proof to clear the Swedish samples from the character of adulterated butters.

And now with regard to the title of Mr. Allen's paper, which appears to me singularly incomplete. If in the syllabus of the meeting of the Public Analysts for December, there had been the slightest indication that the abnormal butter had been Danish or Swedish, I think it probable that very valuable information might have been obtained on this subject, as Dr. Bell, Mr. Betley, of Wigan, and myself, one or all, would in all probability have been present.

And now for the main object of my paper, which is to demonstrate that butter analysis is as reliable as ever, and that so-called abnormal samples of butter have not as yet been proved to be anything more than mixtures.

One cannot help being struck by the phenomenal breed of Danish and Swedish cows, which, according to the information obtained by Mr. Allen, secrete one week perfectly good butter-fat, and without any notice, and without change of conditions another week produce what an experience of all British butter would lead us to call a mixture.

If this is possible a very serious injustice has been done to the Danish agriculturist by those who are acting in his defence.

The so-called supervision of the milking at these Danish farms was performed by gentlemen whose honour, intelligence, and high ability in the special professions to which they are devoted cannot be questioned. On the ground, however, of their entire lack of experience of the new calling which they have undertaken, I think grave objection might well be taken.

There were five gentlemen to supervise the milking of sixty-five cows at B farm.

Mr. Allen shows exactly the amount of supervision he, with his experience, thought necessary, when he says, the milking, etc., was conducted under the unremitting supervision of the whole party, "*especially* of Mr. McGregor and myself."

If the party of five could by any possibility have seen sixty-five cows milked *without losing sight of any portion of the milk for an instant* until it was sealed up, then why is it necessary to weaken the effect by singling out as deserving of special remark, "especially Mr. McGregor and myself"?

The fact is, I fear, there is not one of these gentlemen who would venture, under cross-examination in a Court of Justice, to declare that *no* oily matter *could* possibly have been introduced in the milk.

This is especially the case when it is remembered that a very small quantity would if added to the milk make a 20 per cent. adulteration of the butter, indeed one part to 170 parts of milk would suffice. Then, again, the milk should have been creamed at once by the ordinary separator, so that churning could have taken place without leaving the milk all night, which was dangerous, even though it was sealed up. Surely it cannot be alleged that in such well-regulated dairies this apparatus is not known.

From the foregoing remarks it will be evident that fraud might have been committed. I do not assert it was so committed. I do say, however, that the agent of the Danish Agriculturists in England has done the farmer a serious injustice in not completely proving the impossibility of fraud. This is, indeed, very necessary when it is remembered that nearly all the mixtures which have been condemned in this country have been alleged to be Danish or Swedish, and I trust the Local Government Board or the Board of Trade will cause a complete investigation of the whole question to be made at an early date, so that analysts may reach finality in their inferences from butter analysis.

I have only one word more to add, it is in connection with Mr. Allen's apparent wish to show that the specific gravity of B and O are normal.

This is effected by finding fault with our old friend Westphal, which instrument, since I first introduced it for ascertaining the specific gravity of liquids at high temperatures, has been of great value to the analyst.

Mr. Allen should have given figures for, say, beef-fat taken under the same condition as butters B and O, using the faulty division eight, of the Westphal. I give them below, so that it may be seen that there is a good margin by the gravity process between butter-fat and ordinary fat, viz.—

Butter O, specific gravity at 212°	866·3
Beef-fat, specific gravity at 212°	859·6

It would also have been most valuable information if Mr. Allen could have given the gravities of beef-fat and butter-fat by the specific gravity bottle used by Professor Stein.

One word more regarding the Reichart method, which has always met with such favour at the hands of Mr. Allen, and which I am informed is the one almost, if not exclusively, relied upon by Professor Stein for his daily examination of butter. I desire to call attention to the paper by Dr. Wollny in the January and February numbers of the ANALYST for 1888. In page nine, experiment 135, Dr. Wollny gets 20·45 c.c., as the Reichart of a genuine butter. On page ten, the explanation of this extraordinary result is given, and this explanation together with the possible sources of error (one possible error is, we may get minus 30 per cent. from cohesion of fatty acids) which Dr. Wollny points out upon page forty, would be sufficient to cause us even now considerable doubt as to the reliability of the process.

In conclusion, the inference, I think, which may fairly be drawn from the foregoing remarks is that the specific gravity method either of Dr. Bell or myself, and the Angell-Hehner insoluble and soluble fatty acid determination, as modified by Dr. Muter and others, are still the most reliable methods for determining the genuineness of butter.

Collected Details of so-called Abnormal Butters.

	Somerset House.				Estcourt.				Allen.			
	Specific Gravity.	Insol. Fatty Acids.	Sol. Fatty Acids.	Reichart.	Specific Gravity.	Insol. Fatty Acids.	Sol. Fatty Acids.	Reichart.	Specific Gravity.	Insol. Fatty Acids.	Sol. Fatty Acids.	Reichart.
Danish Butter, 1884. 1. ..	Not given.	89·35	4·18		At 210° F. 865·70	89·45	4·20					2·5 grms. Butter.
,, ,, ,, 2. ..		88·85	4·24		866·00	88·59	4·31					
Irish Butter, 1885... ..	.. ..	..	..	..	865·10	92·07	..	..	..	..	..	7·60
Danish Butter, 1885. ..	908·42 at 100° F.	90·08	3·48		862·50	90·12	3·50	..	..	..	..	8·9
									Prof. Stein.			
Swedish Butter, 1888. Wigan, No. 402. ..	909·4 ,,	90·00	3·32		At 212°. 865·5	89·80	3·40		..	..	..	24·0
Swedish Butter, G, 1888. ..	.. ..	..	..	..	864·5	89·7	3·53	23·60	866·0	..	..	24·2*
,, ,, N, 1888. ..	.. ..	..	..	..	864·0	89·0	3·57	22·55	866·0	..	..	23·9*
									Allen.			
Danish Butter, B, 1888.	909·9 ,,	90·00	4·50		Not enough of the Sample.	90·2	4·51	22·44	864·0	90·43	4·44	5 grms. 22·39
,, ,, O, 1888.					866·30	89·8	4·70	24·64	864·10	90·10	4·67	24·70

Note.—At my request, made on reading the paper upon "Abnormal Butters," Mr. Allen promised to let me have samples of B and O, if any portion was still left. Accordingly, I received an original sealed sample of O, and a small quantity of the rendered fat of B.

ON SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. Atwater.

(Continued from page 18.)

Five series of trials were made. Previous experience had persuaded us that when (1) the tubes are of moderate length and compactly filled with soda-lime, (2) only a moderately high heat is used, (3) the combustion does not proceed too slowly, and (4) the flames are turned off and the tube allowed to cool slightly before aspirating with air, there is no considerable loss of nitrogen by either dissociation or oxidation. In the first series, in which the operation was conducted in the usual way, a confirmation of this impression was sought. In the second series the effect of increasing the length of the tube and with it the time of sojourn of the ammonia gas in the tubes, without increasing the heat, was observed. In the third series the effect of high heat was tested by using long tubes as in the second series, and making the heat much greater. In the fourth series it was sought to learn whether the loss of nitrogen observed in the third series was due to dissociation or oxidation or to both. The trials of the fifth series had a similar object. The results are given in detail in Table II., which includes all the determinations made, and are recapitulated in Table III.

The figures of the first series indicate that with tubes of our ordinary length, 40 cm. or a little less, compactly filled (about 16 cm. with mixture of fine soda-lime and substance, 4 cm. with "rinsings" of fine soda-lime, and 12 cm. with an anterior layer of coarse soda-lime), at a heat sufficient to make the tube dull red, designated here as "medium," and with aspiration by air after the tubes had begun to cool, there was no loss of nitrogen.

In the second series the tubes were long enough to make the anterior layer of coarse soda-lime about 35-40 cm.; the charging otherwise was the same as in the first series. The percentage of nitrogen obtained was the same. Lengthening the tube had no effect upon the result. At moderate heat there was no loss by dissociation with closely-packed tubes, even when the latter were very long and the exposure of the ammonia to the heat was increased.

Table III.

Recapitulation of Determinations of Nitrogen in Ammonium Sulphate under Different Conditions.

Series.	Length of Tube and Anterior Layer of Soda-lime.	Heat of Combustion.	Aspirated with.	Nitrogen found.		Loss of Nitrogen in per cent. of total Nitrogen.	
				Average.	Range of Variation.	Maxim'm.	Average.
				Per cent.	Per cent.	Per cent.	Per cent
1st	Usual.*	Medium.	Air.	21·12	0·03	..	..
2nd	Very long.†	Medium.	Air.	21·12	0·01	..	..
3rd	Very long.†	High.	Air.‡	20·83	0·06	1·5	1·4
4th	Very long.†	High.	Hydrogen.	20·83	0·21	2·0	1·4
5th	Very long.†	Medium.	Hydrogen.	21·11	0·03	..	..

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* Tube about 40 c.m., anterior layer of soda-lime about 12 c.m.

† Tube 75 c.m., anterior layer about 35-40 c.m.

‡ In one case with carbonic acid.

In the third series the conditions were the same as in the second, except that the temperature was made as high as the tubes of the most difficultly fusible Bohemian glass we could obtain would endure without bursting. The standard acid employed was rather concentrated, so that but a small quantity was needed in the nitrogen bulb, and the internal pressure was thus reduced to a minimum. It was thus possible to heat the tubes to bright redness without their bulging. Instead of 21·12, only 20·83 per cent. of nitrogen was obtained. This falls short of the actual amount by 0·29 per cent. of the weight of the sulphate of ammonia, or 1·4 per cent. of the total nitrogen. The indication of loss by either decomposition or oxidation of ammonia was very apparent.

In the fourth series the conditions were the same as in the third, except that air was excluded, the object being to find, if practicable, how much of the loss in the previous series was due to the burning of ammonia by the oxygen of the air, and how much to dissociation. In No. 10, the length of the tube was actually 80 cm. instead of 75 cm. as in the other cases. The extra 5 cm. of the posterior end were filled with bicarbonate of soda, which was heated at the beginning of the experiment to expel the air present in the tube, and again at the end to drive out the residual ammonia. In Nos. 11 to 14, as in all of the next series, in which hydrogen was used for the same purpose, the tubes were about 80 cm. long or a little longer, the extra 5 cm. at the posterior end being filled with asbestos. The hydrogen, which had been dried by sulphuric acid, was passed through the combustion tube for three quarters of an hour, and the tube was then exhausted by a mercury pump, after which hydrogen was again passed through for half an hour. Assuming that the air had thus been very nearly all removed, the combustion was conducted at the very high heat, as in the third series. When it was done, hydrogen was again passed through to wash out the residual gases, precautions being taken to admit no air. The amount of nitrogen obtained averaged the same as in the third series, 20·83 per cent. The exclusion of air made then no difference in the result. This indicated that the loss was due not to oxidation, but to dissociation of ammonia.

It is observable, furthermore, that the individual results in the third and fourth series vary considerably, the range in the fourth series being ·21, while in the first and second series it was only ·03 per cent. While the agreement of duplicates is a very uncertain evidence of the correctness of an analysis, a wide disagreement may be regarded as a tolerably sure indication of error.

In the fifth series the conditions were the same as in the fourth, except that the combustion was made at "medium" heat. The results, which average 21·11 per cent., are practically identical with each other and with those of the first and second series. The difference in condition between this and the fourth series, like that between the second and third, is simply one of heat, and that difference in each case evidently makes the whole of the difference in result.

To resume:—In every case in which the medium heat was employed, whether with long or with short tubes, whether with or without air, the full amount of nitrogen was obtained. In every case with the high heat there was a loss from 1 to 2 per cent. of the total nitrogen in the substance. Neither the length of the tubes nor the presence or absence of air made any difference in either the amount of nitrogen obtained or the uniformity of the results, but the high heat materially reduced the amount of nitrogen and made wide variations in results of duplicate determinations.

That more definite statements as to temperature at which the dissociation takes place are to be desired, goes without saying. Ramsay and Young,* in a series of very interesting experiments on the temperature of dissociation of ammonia, find that the point at which it commences varies greatly with the nature of the surrounding medium.

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* *Jour. Chem. Soc.*, 1884, Proc. p. 88.

In a porcelain tube filled with broken pieces of porcelain, in an iron tube filled with porcelain, and in a glass tube filled with ignited "porous" asbestos cardboard, to expose the gas to a large surface, the decomposition commenced at about 500° or below. At 500°-520°, however, it was very small, but increased gradually at higher temperatures. In contact with a glass surface the temperature at which decomposition begins was much higher. The nature of the surface heated has a very great influence on the amount of ammonia decomposed. In a plain dry iron tube the decomposition was apparently complete at 780°, but if water was present only 95 per cent. was decomposed. At 760°, in a glass tube containing iron wire, 64·3-76 per cent. of the ammonia was decomposed; but when copper was substituted (the other conditions remaining essentially unaltered) only 2·0 per cent. was decomposed. The authors find that "the amount of decomposition depends partly upon the rate of passage of the gas, or, in other words, on the time of exposure to heat," and that "the extent of surface also influences the amount of decomposition.

(*To be continued.*)

LAW NOTES.

Heavy Penalty for Adulterated Milk.—At Wolverhampton Police Court on January 30th, before Mr. Neville (stipendiary)—F. W. Vickers, Tissington, Ashbourne, Derbyshire, was charged with selling adulterated milk.—Mr. Brevitt (town clerk) prosecuted, and said that the difficulty the prosecution had had in this case was to secure the person who, under the Act, was really liable for the alleged adulteration, and, if the defendant were convicted, he must ask for a substantial penalty.—Inspector Blanton deposed that on January 2nd, from information received, he went to the London and North-Western Railway station at Wolverhampton, and saw a churn of milk containing twelve gallons. It bore a warranty label, and was consigned from Ashbourne to the Midland Dairy Company, Horseley Fields. Witness took a sample from the churn, and had it analysed by Mr. E. W. T. Jones, the borough analyst, who certified that it contained 19 per cent. of water.—Mr. Paine, for the defence, urged that the defendant had received no notice from the prosecution of their intention to test the milk, nor had he received a sample from them.—The Stipendiary said he thought it would be fairer to the defendant in future if a sample were supplied to him after it had been taken by the prosecution, although *there was no legal obligation upon them to do so.* It was satisfactory that the original vendor of the milk had been found and proceeded against. In justice to the milksellers in the town, he must impose the full penalty under the Act—namely, £20, and costs.

Adulterated Pepper.—At Glasgow Sheriff Court on the 14th ult. Matthew Kerr, grocer and provision merchant, 61A, King Street, Calton, was charged before Sheriff Balfour, at the instance of Mr. Peter Fyfe, sanitary inspector, with having, on 19th December, sold to Mr. Robert Inglis, assistant sanitary inspector, a quarter pound of black pepper, which on analysis was found to contain 3 per cent. of sand, 4 per cent. of mineral matter, and 7 per cent. of pepper husk, all of which are extraneous to whole pepper. The respondent pleaded guilty. His agent, Mr. Wm. Shaw, writer, stated that Mr. Kerr bought the pepper from a house in Glasgow which held a guarantee from a Liverpool house that the pepper was pure. It was what was known as Penang pepper, which had naturally a large amount of impurity in it. After hearing a further statement from Mr. Shaw, Mr. R. G. Ross, writer, who prosecuted, and Dr. Tatlock, the analyst, his Lordship gave judgment. The Sheriff said that he thought Mr. Kerr had been well advised in tendering a plea of guilty. It appeared that black pepper had got a core and an outside husk, and in preparing it for the market there must be to a greater or a lesser extent a certain quantity of sand and mineral matter adhering to the pepper. Dr. Tatlock had taken the Somerset House standard, which allowed a percentage of 3½ of sand, and in regard to mineral matter he had been guided by public analysts and writers of authority, who allowed 7 per cent. for mineral matters, and as long as the percentage of sand and mineral matter only came to 3½ and 7, no prosecution would be directed against any party who sold black pepper. But the line must be drawn somewhere. From the manner in which this black pepper was prepared, it was perfectly clear that the pepper berries, after being dried, must have particles of earthy matter adhering to the husks, and if black pepper were sent over to this country with an immense proportion of earthy matter adhering to the husks, with the intention of being sold in that way, it would not do to allow such a sale to take place, because the earthy mattter might get in, not by fair, but by foul means. To prevent that the Somerset House and those public analysts and writers of authority had fixed what he presumed were reasonable and fair standards. Dr. Tatlock had acted most liberally in stating the adulteration, because, in point of fact, the mineral matter really amounted to about 16 per cent., and the sand to about 6 per cent. It was not to be supposed that in inflicting a light penalty, as he was going to do, he by any means countenanced black pepper being sold in the state in which Mr. Kerr had sold it. But he had no reason to doubt Mr. Kerr's *bona fides* in the matter. He apparently bought from a wholesale house, who, on their part, held a written guarantee from the foreign sellers. Mr. Kerr, unfortunately, had not been armed with such a written guarantee, and he was therefore liable to the authorities for selling the adulterated article. As it was the first case of the kind that had been brought before him, the Sheriff, in the circumstances, inflicted a penalty of 15s. The fine was paid.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

SIR,—As few penalties for adulteration are inflicted, the enclosed report* may interest your readers, especially as the case comes under the special provision of the Act for procuring milk for analysis at the railway station, or in course of delivery. The following are the analytical results of this sample of milk:—

Specific Gravity at 60° F.	1025·7	
Total Solids	9·93	per cent.
Fat (by Adams' Coil method)	3·03	„
Solids not Fat (by difference)	6·90	„
„ „ (by Bell's method)	7·29	„
Ash	0·63	„
Lactose ($C_{12}H_{22}O_{11}$) by Polariscope	3·60	„
„ „ by Fehling	3·54	„

Yours truly,
E. W. T. JONES,
Public Analyst for Staffordshire, etc.

A MARE'S NEST.

SIR,—Now that the Society of Public Analysts have appointed a *Select* Committee (which for brevity's sake we shall term the A B C of the Society) to investigate the subject of Butter Analysis owing to the supposed alarming state of matters brought about by Mr. Allen's late paper, entitled "Abnormal Samples of Butter," it may be of interest to the members of the A B C to know that as far back as the year 1884, Professor Wiley of Washington, in analysing nine samples of genuine butter found as a minimum for insoluble fatty acids 88·50, and a maximum of 89·89 per cent., the first figure being Angell and Hehner's highest figure.

In the year 1886, Professor E. Waller, of New York, found 90·26 per cent. as a maximum, and 86·40 as a minimum for twenty-six genuine butters examined by him; and in the same year Professor C. B. Cochrin found 87·7 per cent. as his highest figure, and 86·7 as his lowest figure in twenty-five genuine Pennsylvania butters, figures which prove conclusively that it is not only in Danish Butters that high percentages of insoluble fatty acids are to be found. The figure 89·89 is as near as a toucher to 90 per cent. (and we all know what as near as a toucher means), a figure obtained in the year 1884, the difference between 89·89 and 90 being less than the difference between the figures obtained by Mr. Allen in his two analyses which were performed in duplicate, and by a strange coincidence Professor Waller's highest figure 90·24 obtained in 1886 coincides with one of Mr. Allen's estimations.

Is it possible that the A B C are ignorant of these facts?—Yours, etc.,

THE GREAT I AM.

TO CORRESPONDENTS.

F. A. B.—Had you applied some years ago you might possibly have gained admittance without examination, but, so far as we know, such days are now over. We are not aware that there is any relaxation of the rule as to the fixed course of study, and, moreover, we fear that, even if there were, the examination itself would present an insuperable barrier to a man in business who had not time to go through a special course. That this should be so is only just, because it is intended to exclude all but those who really make an actual profession of chemistry as distinct from pharmacy. Undoubtedly some pharmacists have managed to get in at present, but, as they die off, their places are not likely to be filled up by others of the same *status*, because a man who became a practising consulting chemist would naturally drop the shop altogether, it being impossible to combine the two businesses, which are totally distinct in every respect.

PROF. KINNICUTT.—Letter to hand but no paper arrived yet (February 25th). When it comes will make use of it as soon as possible.

Communications on Literary or Exchange Matters to be sent to 325, Kennington Road, London, S.E.

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* *See* Law Notes.

THE ANALYST.

APRIL, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

AN ordinary meeting of the Society was held at Burlington House on Wednesday, 13th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot paper being examined, it was announced that the following were duly elected :—As member, E. W. Voelcker, F.I.C., F.C.S., Analytical Chemist, London. As associate, J. S. Ford, Assistant to Professor Crum-Brown, Edinburgh.

Mr. W. Dibdin, Chemist to the Metropolitan Board of Works, was proposed as a member.

The following papers were read and discussed :—

"Notes on Lactose," by E. W. T. Jones.
"Colouring Matter (Arsenical) Used for Colouring Candles," by W. F. Lowe.
"Methods of Fat Extraction in Milk Analysis," by Dr. Vieth.

A new apparatus for use in Butter Analysis was exhibited and explained by Mr. H. Droop Richmond, and Dr. Vieth also explained the apparatus he uses for the same purpose.

The next meeting of the Society will be held at Burlington House on Wednesday, the 10th inst.

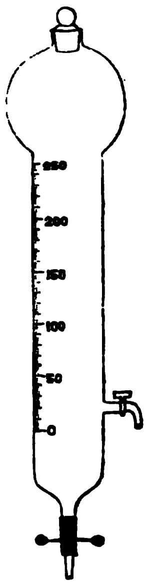
AN IMPROVED METHOD FOR THE ANALYSIS OF FATS AND OILS.

BY JOHN MUTER, PH.D., F.R.S.E., F.I.C., & L. DE KONINGH, F.I.C.

(*Read at the Meeting, February,* 1889.)

INTRODUCTORY.

IN the year 1878 the senior author of this paper contributed to the Society of Public Analysts a method for the estimation of oleic acid in fats and oils. This process was based upon the formation of an absolutely neutral oleate of lead, solution of the salt in ether, and decomposition of the same in a special apparatus by means of hydrochloric acid. The apparatus then described has since been rather extensively used, and is figured in the various price lists as "Muter's Olein Tube." It has since undergone considerable alterations, and, as it plays a prominent part in the method now about to be described, we give, to commence with, a fresh description of the tube as now employed. From the illustration in the margin (which is not to scale, and is too wide in proportion to the length) it will be seen that it is essentially a burette with a bulb at the top, and provided with a well-ground stopper. The graduated part is of such a width as to distinctly show 1 c.c. in the graduations, and it has a stop cock at a height of 50 c.c. from the bottom. The graduations commence at 40 c.c. from the lower end, and continue up to 250 c.c., ending just below the bulb. In winter the tube can be used in a clamp attached to an ordinary retort stand, but when the temperature of the laboratory exceeds 60 F., it may be arranged in an outer jacket, which can be kept filled by running water, or some other means may be taken for keeping the temperature low and fairly equal during the whole progress of an operation. This jacket is not shown in the drawing, but any chemist will easily see how it should be fitted. We are not aware that the improved tube can be got anywhere but from Cetti's, of Brooke St., Holborn (who make them for us); but no doubt they will shortly become as general in the price-lists as the former pattern. In the remarks that follow, this apparatus will be henceforth referred to as the "olein tube," and having thus got quit of any further necessity for referring to it in detail, we proceed to our main subject.



The remarkable advance rendered possible in fat analysis by Hübl's discovery of the iodine absorption method has opened up a large field of research, which is now being actively worked by many experimenters. It will be readily admitted by all analysts that in dealing with the power of fats to absorb iodine, three factors come into play, viz.:—

(1) The difference in the iodine absorbing power of oleic and linoleic acids.

(2) The joint amount of these acids actually present in the fat.

(3) The ability to *always* perform the process in the presence of an *excess* of iodine *that shall be as nearly constant as possible.*

If, therefore, we can first isolate the fluid acids of any fat, and having found the actual amount of such acids present in the sample, submit them to the action of the iodine under fixed conditions and, *without having risked any chance of an alteration in their nature during the process of isolation*, it is evident that we have attained a distinct step in advance of our present methods. Up till now we have been unable to find any published process for the estimation of the fluid acids of a fat without their having been exposed to the air, dried by heat, or a change in their nature otherwise rendered possible by the method of isolation employed. Before such a process could be devised, it was necessary to prove two points. (1) Can the amount of the acids be found directly from their ethereal solution with fair accuracy? (2) Can the iodine be applied to the acids without any previous exposure to the air, and consequently to oxygen? Experiments have proved that both these points are quite attainable. It is t our purpose to overload our paper with a tedious account of such experiments, cause it is a matter that any analyst can easily verify for himself. We will therefore ntent ourselves by saying that a comparison of the results of our process, with those tained by the gravimetric method in the case of fats so widely different in their nature tallow and linseed oil, has proved the sufficient accuracy of the volumetric process reafter detailed, for the purpose for which it is intended. There is little doubt but at the prolonged use of the process, side by side with gravimetric methods, will ulti- ately throw light on the respective combining weights of oleic and linoleic acids, when e venture to think that it will be found that there is not so great a difference as is at esent supposed to exist by many authorities. All such speculations are, however, reign to the purpose of our present preliminary paper, and must be reserved for a ture occasion. At the moment, our intention is simply to give our method of working, d to leave the consideration of its various possible applications to future papers. The bject of the analysis of lard and butter being very pressing matters at present for blic analysts, we are dealing with them first, and will give the results of such investi- tion after describing the details of the main process.

THE PROCESS.

A flask of suitable capacity is counterbalanced, and about 3 grms. of the oil or fat ving been introduced, the weight is ascertained. To this are added 50 c.c. of alcohol d a fragment of solid potassium hydroxide sufficiently large to saponify the fat. The outh of the flask having been closed by a cork, through which passes a long tube drawn t to a fine point, the whole is heated on the water bath until the fat is thoroughly ponified. A drop of alcoholic solution of phenol-phthalein having been added, acetic id is dropped in until the solution becomes faintly acid, and then alcoholic potash is refully stirred in until the *very faintest* pink tinge is restored. With certain oils the lution will be too dark for the use of the indicator in this manner, and in such cases must be applied in the form of phenol-phthalein paper. 200 c.c. of boiling distilled ter are then placed in a 500 c.c. basin, 30 c.c. of a 10 per cent. solution of plumbic etate are added, and the whole brought to the boil. Into this the soap solution from e flask is slowly poured, with constant stirring, and the whole is allowed to cool slowly, rring well so as to cause the precipitate to agglomerate, and the clear liquor is poured f. Boiling water is immediately poured on, and the precipitate is rapidly washed by cantation. When the washing is complete, the precipitate is scraped from the basin to a stoppered bottle, and 80 c.c. of *redistilled* ether having been poured upon it, the maining particles of the precipitate are washed from the basin into the bottle with her, and the bulk of ether in the bottle is finally made up to 120 c.c. The bottle is curely closed and allowed to stand for twelve hours (with occasional shaking), during ich time the plumbic oleate will entirely dissolve. A funnel with a ground edge is en arranged over the "olein tube," a filter is placed therein and the contents of the ttle having been filtered into the tube, the insoluble plumbic stearate, etc., remaining the filter, is washed with ether until the washings pass free from lead. This will ually be attained by the use of 100 c.c. of ether, and during the filtration and wash- g the funnel must be kept covered by a ground glass plate. The funnel having been moved, dilute hydrochloric acid is poured into the tube up to the first mark (thus using about 40 c.c. of acid, 1 in 4), and the tube having been closed by the stopper and taken from the clamp, is thoroughly shaken until the decomposition is complete, which is indicated by the ethereal solution clearing up. The tube having been put back in its support, the liquids are allowed to separate and the aqueous layer is run off by the bottom pinch cock. Water is then poured in up to the same mark, the whole is again shaken and separated as before, and this is repeated until the washings are drawn off free from acidity. Water is then once more run in until the ether is forced up to the zero mark, and ether is added (if necessary) so as to bring its upper layer to a definite point (say 200 c.c.), and the total volume is read off and noted. 50 c.c. of the ethereal solution of the fluid acids are run into an Erlenmeyer's flask, and the flask having been attached to a condenser, the ether is distilled off by a bath of warm water until only a little remains. It is important that the whole of the ether should not be distilled off, so as to avoid contact of the acids with the air. 50 c.c. of pure alcohol (or methylated spirit that has been rectified over potassium hydroxide) are added to this residue, and the solution is titrated with deci-normal soda, using phenol-phthalein as indicator. Each c.c. of soda used represents ·0282 of oleic acid, and the amount found is calculated up to the total bulk of the ethereal solution, so as to obtain the total fluid acids in the weight of fat started with.

Having thus ascertained the total acids, and also the strength of the remaining ethereal solution of the same in the "olein tube," the next step is to run off as many c.c. of that liquid as will contain ·5 grm. of oleic acid (or as nearly that quantity as can be conveniently measured by means of the instrument, of course carefully noting the amount taken) into a stoppered bottle of at least 350 c.c. capacity, and to immediately close the mouth of the same by a cork through which passes two tubes, the one going down nearly to the surface of the liquid, while the other just passes through the cork. The long tube is then connected with a gas apparatus, in which carbonic anhydride* is being generated from marble and hydrochloric acid, and the gas washed by passing it through a solution of sodium bicarbonate, and then dried by passing it over fused calcium chloride. The bottle is placed in water at about 120° F., and a rapid stream of gas is passed through until *every trace* of the ether has evaporated. To the residue 50 c.c. of *Hübl's reagent* is instantly added, and the stopper having been at once inserted, the bottle is put aside in an absolutely dark cupboard for twelve hours. A blank experiment is, as usual, started in another similar bottle with the same amount of Hübl's reagent, and set to stand side by side with the other bottle. At the expiration of the proper time, 35 c.c. of 10 per cent. solution of potassium iodine are added to each of the bottles, and the contents having been made up to 250 c.c. with water, 15 c.c. of chloroform added, and the whole shaken, both are titrated in the usual manner with a deci-normal solution of sodium thiosulphate (hypo) that has been just previously standardised with an accurate deci-normal iodine solution. The difference between the amount of "hypo" consumed by the check and the experiment respectively, gives the amount of iodine absorbed by the weight of oleic acid taken, which is then calculated to the amount required by 100 grms. and put down as the "iodine absorbing power" of the fluid acids of the fat under examination.

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* NOTE.—When the paper was read it was stated that ordinary gas had been successfully employed, but since that we have preferred the arrangement now detailed. It is very important that the volume of the ether should not alter between the running off of the first 50 c.c., and the taking of the amount representing the ·5 grm. of acid. If it cannot be maintained by regulating the temperature, the volume remaining after taking the 50 c.c. must be noted, and then again renoted before taking off the second quantity, and corrected by calculation.

Such therefore is the process, and by it we not only obtain the amount of fluid acids present in the fat with a fair amount of accuracy, but we also believe that a reasonably constant iodine number for the acids from each fat can be determined when in a reasonably fresh condition, thus enabling us to calculate the amount of any admixture with a very much more tolerable amount of certainty than has hitherto been attainable. We invite our colleagues to try the method, and to assist us in establishing reliable iodine numbers for the acids from every oil and fat. Meantime we are going on with such an investigation, and will continue to submit the results, as rapidly as possible, in the pages of the ANALYST. As being at the moment the most interesting to public analysts, we have commenced with some solid fats used for food.

(*To be continued.*)

DENSITY.

By H. Droop Richmond.

(*Read at Meeting, February,* 1889.)

When we talk of the density of a body we mean the mass of a unit of volume of that body, or, as the unit of mass has been fixed at 1 grm., and the unit of volume at 1 cubic centimetre, the number of grms. in 1 cubic centimetre; thence it follows that density is not a relative, but an absolute property. Notwithstanding that the cubic centimetre has been fixed as the volume of 1 grm. of water at 4° C. (the point of maximum density), a custom has sprung up of assuming that an instrument for taking density graduated to hold x grm. of water at 15°, 15·5°, or 16° C. (as the case may be) holds x cubic centimetres; this arbitrary creation of units of volume, besides being clearly wrong, is liable to create confusion, and renders the expression of results uncertain, which uncertainty is not removed entirely by the use of such an illogical expression as "the density at 15° compared with water at the same temperature." To compare an absolute quantity is absurd; this uncertainty is much less, however, than that caused by errors of observation, many of which are not even noticed, much less corrected.

I do not propose to discuss either the taking of densities of either gases or solids, on account of the special methods and precautions against errors, which are so well known in the case of gases, and of the comparative unimportance attaching to the determination of the densities of solids; my remarks must therefore be taken as applying to liquids only.

We use practically three methods for determining the densities of liquid:—1. By hydrometers. 2. By the measurement of the loss of weight of a solid in a liquid, *e.g.*, the Westphal balance and the torsion balance. 3. By the determination of the mass of known volume, *e.g.*, the pyknometer.

1. Hydrometers.—The faults of these instruments consist in errors introduced by the cohesion of the liquid to the material of which the instrument is made, which render it difficult to read off the exact point which is at the level of the surface of the liquid; as also the volume of the portion immersed is determined in part by the cohesion, it follows that hydrometers do not give the same reading in different liquids, whose density may be the same, if the viscosity is different. Another error is caused by expansion by heat if the hydrometer is used at a different temperature than that for which it was graduated. Their merits consists in speed, and if they are graduated for

use in one particular liquid, and at a pretty constant temperature, fairly exact results are obtainable (within ·0005).

2. Instruments which measure loss of weight in a liquid.—Of these the Westphal balance is the most important and widely used, and is extremely quick and convenient, where great accuracy is not required, though open to grave errors. Its faults are the errors due to cohesion (very slight); to the difference between true weight and apparent weight in air; to the condensed film of air on the plummet, which is, however, removed on immersion in most liquids; to the unequal expansion of plummet and liquid of which the density is to be taken at any other temperature than that for which the instrument is set, and of the arms of the balance; to the wire or thread used to suspend the plummet (usually reduced to a minimum by the use of a very fine thread); and to the imperfections in the balance (often large in those commonly in use). Many of these errors may be neglected in ordinary work, for instance, those due to cohesion, unless the fluid is very viscous; to difference between true weight and apparent weight in air, if the substance has a density near 1; to the condensed film of air; to the use of a wire or thread; and to the expansion of the arms. It is in the construction that the Westphal balance chiefly errs, one error having been pointed out by Allen (ANALYST, 1889, p. 11) in the length of the intervals between the divisions. The beam of the Westphal balance is usually made 10 centimetres in length, so that to insure absolute accuracy the intervals should be 1 centimetre apart, within $\frac{1}{100}$ of a millimetre, an amount of accuracy which it is not easy to attain in practice, and which may easily be diminished by wear. Another error is introduced in the hook riding on a knife edge, from which the plummet hangs; if the position of this is changed, as it must necessarily often be, a difference in the reading may be observed, amounting sometimes to ·001. Another error may occur from one of the riders not sitting absolutely exactly in its place, but inclined to one end of the beam or the other; this error may amount to as much as ·0005. The error introduced by the expansion of the plummet is of course obvious, and should be allowed for by noticing the difference between the actual density of water at various temperatures and the indicated density. Unless this is done, determinations made at higher temperatures are only relative, and cannot even be compared with those obtained by another instrument, unless the coefficient of expansion of the material of which the two plummets are made is the same. An inconvenience is felt if the density of a liquid is to be taken at a temperature differing from the temperature at which it is a comparatively large bulk of liquid having to be warmed or cooled as the case may be. I should fix the limit of accuracy of the Westphal balance at not much less than ·0005, although closer duplicate readings are often obtained.

The torsion balance is open to all the faults of the Westphal balance, except those due to construction, and besides has the tendency to acquire a slight permanent set, and is not to be recommended except for rough density determinations.

3. Instruments in which the mass of known volume is taken, or pyknometers.—These are practically two in number, the specific gravity bottle and the Sprengel tube and its modifications, neither of which presents any real advantage over the other. It is perhaps rather easier to adjust the temperature of the liquid in the Sprengel tube, but, on the other hand, the Sprengel tube is more liable to lose weight than the bottle, when in constant use. The errors, all of which can be readily allowed for, are the following: those introduced by the condensed film of air, both inside and outside; by the difference between true weight and apparent weight in air; and by the expansion of the glass or other material of which the instrument is made. The most difficult to allow for is the condensed film of air, which I consequently try to eliminate as far as possible by the following plan: dry the bottle or tube by heating it and passing a

urrent of dry air through it (of course removing the thermometer if the temperature higher than the highest it will bear), cool, by pouring a current of cold water on the utside, wipe dry and weigh at once; duplicate weighings very rarely differ by as uch as ·0005 grm. I then fill with distilled water freshly boiled in a platinum vessel, djust the temperature accurately to 4° C., wipe dry and weigh at once. The increase f weight in grams., corrected for the volume of displaced air, will give the number of cubic entimetres the bottle or tube will hold. The difference between duplicate weighings hould not exceed ·0005 grm.; the difference between the capacity at 4° and the weight orrected) of water in grms. held by the bottle or tube at any other temperature, ivided by the density of water at that temperature, will give the increase of capacity ue to expansion of the glass or other material of which the bottle or tube is made. A ble of capacities at various temperatures, and a formula for the expansion, may easily e made of it for each instrument.

TABLE OF DENSITIES OF WATER.
(Compiled from the results of Pierre, Despretz, and Kopp.)

Temp. C.	Density.	Temp. C.	Density.
0°	·99988	40°	·99237
4°	1·00000	50°	·98817
10°	·99975	60°	·98343
15·5° (60° F.) ..	·99910	70°	·97794
20°	·99826	80°	·97193
30°	·99575	90°	·96561
37·8° (100° F.) ..	·99313	100°	·95865

Should the capacity of the instrument be required at 100° C., some rather special recautions must be taken; the tube should be filled hot, and while the water is bsolutely boiling, and the liquid should be adjusted to the proper level, while under ight pressure. I accomplish this as follows (with a Sprengel tube): To the end of e tube from which the liquid is drawn, a tube, about 1·2 c.m. in diameter, having its her end narrowed, and to which a side tube is attached, is fixed by means of a cork; a d, on the end of which a bundle of filter paper or other absorbent material is fixed, is ssed through the narrowed end in such a way that the joint is air-tight (*i.e.* either rough a cork or india-rubber); the side tube, and the other end of the Sprengel tube e attached to a bottle, by means of a T-piece, in which a pressure of about ten inches mercury is kept up by suitable means; the Sprengel tube is, after being filled, im-ersed in steam, and when the water ceases to expand the rod supporting the filter-per, is brought to the end of the tube and by its means the water is accurately adjusted the mark; the tube is then taken out and weighed in the usual manner.

The determination of densities at higher temperatures than 15·5° is usually only quired for body that are solid at that temperature, such as butter, and other fats; uter proposed to do this at 100° F. (37·8° C.) and graduated his instruments with ater at the same temperature, and gave to these determinations the name of "actual nsity," an unfortunate name, as they were really the actual densities divided by the nsity of water at that temperature. These, however, were good determinations, as ere could be no mistake as to what they really were. Three years ago Estcourt oposed to take density of fats at 100° C. by the Westphal balance, a plan which has come rather general among English analysts, but is a distinct falling off from Muter's ethod, because the expansion, which is not usually allowed for, is enormously increased

at that temperature; and moreover the temperature is exceedingly difficult to obtain, as the boiling point of water depends on the pressure of the air. I do not consider that the results obtained by two analysts taking their densities thus are comparable within ·002; the only advantage which this method has over the other, is that beeswax and carnauba-wax are liquid at 100° C. while solid at 100° F. In the regulations for analysis for the State of Colorado, it is laid down the densities of fats shall be taken at 40° C., which seems to me the most convenient temperature, now that the Centigrade scale is in general use, and I think that we may expect, if the precautions I have drawn attention to are taken, that two analysts should not differ by more than ·0002 or ·0003.

There are two minor precautions I should wish to draw the attention of analysts to, first, to allow a sufficient time (when using the bottle or tube), for the liquid to contract to its full amount; alcohol contracts very quickly, glycerol very much more slowly, while as an extreme case Perkin has recorded a mixture of aldehyde and water $C_2H_4O + H_2O$ which took several hours (*J. Chem. Soc.* 1887, p. 817); the other precaution is to use an accurate thermometer, the great majority of low-priced thermometers being sensibly wrong.

In conclusion, I would recommend that all densities should be the weight (mass) of the substance in the cubic centimetre, that the density of liquids should be taken at 15°, and of fats at 40°. A symbol, such as $D_{15°}$ meaning density at 15° and $D_{40°}$ at 40° might be advantageously adopted.

Some results actually obtained are appended.

I. Experiments showing the unequal expansion of specific gravity bottles and tubes:—

Vol.	Between 15·5 and 37 8	Expansion per c.c. of capacity.
25· c.c.		·00042
12·1 c.c.		·00055
7·9 c c.		·00044
5·9 c.c.		·00081

II. Results obtained in graduating a Sprengel tube:—

Wt. of Sprengel after heating and rapid cooling	**18 6308 gr.**
" " after standing 15 min. . .	**18·6328 gr.**
Difference due to condensed air film	**·0020 gr.**
Wt. of Sprengel filled with water at 4·0° . .	**30·7622 gr. 30·7616 gr.**
" " " " 15·5° . .	**30·7523 gr. 30·7525 gr.**
" " " " 30·0° . . .	**30·7158 gr. 30 7162 gr.**
" " " " 37·8° . .	**30·6865 gr. 30 6875 gr.**

Capacity of Sprengel:—

	Fund.	Calc.
4°	12·1448 c.c.	——
10°	12·1454 c.c.	12·1454
15·5°	12 1462 c.c.	12·1462
20°	12·1476 c.c.	12·1475
30°	12·1505 c.c.	12·1506
37·8°	12·1535 c.c.	12·1533
40°	——	12·1541

Formula for capacity at *t*°:–

$$V = 12{\cdot}1448 + {\cdot}0001(t-4)^{\circ} + {\cdot}0000045(t-4)^2$$

ON THE COMPOSITION OF MILK AND MILK PRODUCTS.

By Dr. P. Vieth.

(*Read at Meeting February,* 1889.)

I have to bring before you to-night is my annual report on the work done g the year 1888 in the laboratory, which is under my charge. Particulars with l to the purpose for, and the way in which the work is carried out may be found r former papers of a similar nature (see the Analyst, VII., p. 53; VIII., p. 33; . 56; X., p. 67; XI., p. 66; XII., p. 39; and XIII., p. 46), and need not be ed on the present occasion.

The total number of samples submitted to analysis during the year 1888 is 20,248, ising—

18,354	samples of	milk.
1,144	„ „	cream.
553	„ „	skim milk.
26	„ „	buttermilk.
44	„ „	butter and butter-fat.
6	„ „	cheese.
35	„ „	milk preparations for infants and invalids.
59	„ „	water.
27	„ „	sundry articles.

In addition to these analyses, the specific gravity of about 75,000 samples of whole kim milk was determined.

Of the milk samples 12,682 were taken from the railway churns on their arrival dairy, and 2,545 by the inspectors employed by the business, from the men while atter were serving the customers. The following table contains the monthly ges of the results referring to these samples:—

Average Composition of Milk.

1888.	Samples Taken				
	On Arrival.				On Delivery.
	Specific Gravity.	Total Solids.	Fat.	Solids not Fat.	Total Solids.
ry ..	1·0325	12·97	3·79	9·18	13·0
ary ..	1·0325	13·00	3·81	9·19	13·0
h	1·0325	12·90	3·73	9·17	12·9
.. ..	1·0324	12·81	3·68	9·13	12·9
.. ..	1·0324	12·82	3·69	9·13	12·9
.. ..	1·0324	12·83	3·69	9·14	12·9
.. ..	1·0320	12·82	3·76	9·06	12·9
st ..	1·0319	12·84	3·80	9·04	12·8
mber ..	1·0322	13·06	3·94	9·12	13·0
er ..	1·0325	13·09	3·89	9·20	13·0
mber ..	1·0322	13·18	4·03	9·15	13·1
nber ..	1·0321	13·01	3·91	9·10	12·9
rly average	1·0323	12·94	3·81	9·13	12·9

I may safely leave it to you to draw your own conclusion from these figures.

A further number of 1,806 milk samples was analysed in connection with a very ded investigation into the composition of milk yielded by individual cows kept at the Aylesbury Dairy Company's estate, near Horsham. The results of this interesting investigation, which was commenced in the year 1887, will form very valuable material, which I hope to be able to lay before you on some future occasion.

As in the case of milk, so in the case of cream, supplied to customers, two series of samples were examined, viz., 412 samples taken before the cream was sent out, and 304 samples taken by the inspectors from the men when working their rounds. The results were as follows:—

AVERAGE AMOUNT OF FAT IN CREAM.

1888.	Samples Taken	
	Before Sent out.	On Delivery.
January	43·8	44·7
February	43·4	43·5
March	44·1	44·4
April	45·0	45·1
May	45·5	46·5
June	45·4	45·9
July	45·6	45·7
August	45·1	46·0
September	45·6	45·5
October	46·6	46·7
November	48·0	48·0
December	47·2	46·6
Yearly average	45·4	45·7

The agreement between the two series of samples is satisfactory, considering the difficulty of drawing a fair average sample of cream of such richness, and further considering that cream sent out with the morning delivery was sampled before sent out, but not on delivery, being handed to the men, and by the latter to the customers, in sealed cans.

The composition of 55 samples of clotted cream was as follows:—

Water	31·57	to	44·11	average	35·54	per cent.
Fat	45·78	„	61·49	„	57·09	„
Solids-not-fat	5·82	„	10·14	„	7·37	„
Ash	·44	„	·80	„	·57	„

Skim milk produced by abstracting cream from milk by means of the centrifugal cream separator was exceedingly poor in fat, containing generally less than ·3, and only in exceptional cases more than ·4 per cent. of fat.

The butter samples were derived from three different sources; six referred to butter churned in the Aylesbury Dairy Company's dairy, fourteen to French, and fifteen to Danish and Swedish butter. I think it will be more instructive to give the results of the analyses of the three kinds separate.

BUTTER CHURNED IN LONDON.

Water	10·94	to	12·61	average	11·72	per cent.
Fat	85·66	„	87·59	„	86·53	„
Proteids, etc.	·14	„	·73	„	·41	„
Ash	·79	„	2·51	„	1·34	„
Chlorides, as NaCl ..	·68	„	2·30	„	1·20	„
Insoluble fatty acids, Hehner	88·27	„	88·39	„	88·32	,
Deci-normal alkali, Reichert	12·9	„	13·3	„	13·1 c.c.	

FRENCH BUTTER, FRESH.

Water	13·40	to	14·41	average	13·79	per cent.
Fat	83·98	,,	85·58	,,	84·86	,,
Proteids, etc.	·89	,,	1·56	,,	1·16	,,
Ash	·14	,,	·25	,,	·19	,,
Chlorides, as NaCl ..	·05	,,	·12	,,	·08	,,
Insoluble fatty acids, Hehner	87·15	,,	87·55	,,	87·38	,,
Deci-normal alkali, Wollny	26·1	,,	27·6	,,	26·9	c.c.

DANISH AND SWEDISH BUTTER, SALT.

Water	11·78	to	15·65	average	13·72	per cent.
Fat	81·72	,,	85·49	,,	83·11	,,
Proteids, etc.	·71	,,	1·71	,,	1·09	,,
Ash	1·32	,,	2·71	,,	2·08	,,
Chlorides, as NaCl ..	1·12	,,	2·44	,,	1·85	,,
Insoluble fatty acids, Hehner	87·30	,,	88·43	,,	87·78	,,
Deci-normal alkali, Reichert	13·0	,,	14·2	,,	13·6	c.c.
,, ,, Wollny	27·6	,,	29·3	,,	28·3	,,

Two samples of butter-fat, which had become bleached by three years' exposure to e action of air and light, contained 83·79 and 84·18 per cent. of insoluble fatty acids spectively.

The examination of two preparations, sold as preservatives for milk and cream der the names of "Preservitas" and "Neigeine" respectively, revealed the old story, ., that the preparations contained boracic acid as active principle.

In addition to bringing under your notice the above analytical results, I should e to make a few observations on specific gravity determinations.

Although the specific gravity offers such a ready means for the detection of that olesale adulteration which consists in the addition of large quantities of water, and ich has so frequently been, and still too often is, heard of through the police courts' oceedings, it is only of comparatively recent date that attention is paid in this country the determination of the specific gravity of milk. Of late, however, this determina-n has become of particular importance, forming, as it does, in conjunction with the termination of the total solids, the basis for calculating the fat, thereby avoiding, or at y rate checking, the determination of the latter constituent.

To ascertain the specific gravity quite correctly becomes under these circumstances great importance. I am convinced that specific gravity bottle, Sprengel tube, or tometer give, if carefully used, equally satisfactory results, the last-named instrument doubtedly with the least amount of trouble. That due regard must be paid to the nperature is a matter of course, but another precaution may be mentioned in this ce, which, although it has been long, is still less widely known. I refer to the rise of specific gravity which takes place and continues for a considerable time in milk, after latter has been drawn from the udder, and which is quite independent from the escape gases the milk might have contained.

I availed myself of an opportunity which presented itself to make some obser-tions on this point. The experiments referred to milk yielded by cows kept in a London d. The cows were milked between 4 and 5 o'clock in the afternoon, a sample drawn m the mixed yield, and its specific gravity determined at about 5.30 o'clock. The termination was repeated on the following morning, at 9.30. Fifteen samples showed an rage specific gravity of 1·0296 directly after milking, and of 1·0309 after sixteen urs, or an average rise equal to ·0013. In two instances the rise amounted to ·0020, in to ·0015, and in seven to ·0010.

According to investigations made by Recknagel, the specific gravity becomes station-, *i.e.*, normal, after five hours or less, provided the milk is cooled down below 15 C.;

at temperatures above 15° C. the apparent contraction proceeds much more slowly, and goes on for twenty-four hours, and even longer. If milk, after its specific gravity had become stationary, is warmed up to 40° C. the specific gravity will be found to have decreased, and it will take some time before it becomes normal again. Recknagel believes that molecular changes in the state of the casein account for the remarkable phenomenon.

(Conclusion of the Society's proceedings.)

ABNORMAL DANISH BUTTERS: A REPLY TO MR. ESTCOURT.

By Alfred H. Allen.

The article by Mr. Estcourt on Abnormal Butters, published in the last number of the Analyst requires no detailed reply from me, but there are some points which it may be well to explain, lest other readers should have drawn as erroneous deductions as those of Mr. Estcourt. It is to be regretted that Mr. Estcourt did not make his criticism the subject of a paper before the Society of Public Analysts, as the misconceptions into which he has fallen could then have been at once met and explained, and time, space, and trouble saved thereby.

Mr. Estcourt writes of my "apparent wish to show that the specific gravity of B and O are normal." In my paper I distinctly make the statement that the average density of butter fat at 100° C. is ·8658 (corrected). The abnormal Danish butters B and O are described (page 10) as having specific gravities of ·8639 and ·8641 respectively. How, in the face of these figures, Mr. Estcourt can have supposed that I regarded or "wished to show" the specific gravity of these samples to be normal I am unable to understand. The very object of my paper was to show that genuine butter might be physically and chemically abnormal to an extent not generally recognised. Everyone knows that there is a marked difference between the density of beef-fat and that of butter-fat, and this difference will be just as great whether the observations are taken with a faulty instrument or an accurately divided one. Mr. Estcourt himself for years used a Westphal balance the plummet of which was broken, and hence was only capable of yielding arbitrary results, strictly comparable among themselves no doubt, but which were incapable of being published or communicated to other chemists.

Whatever Mr. Estcourt may "fear," I am quite prepared, if need be, to maintain, under cross-examination in the witness-box, that no foreign oily matter *could* possibly have been introduced into either of the Danish milks during the manufacture of the abnormal butters B and O. The produce of each of the 31 cows at B farm was brought as obtained by the milkers, who were fully within view, to a weighing machine, where the yield of each separate cow was observed, and the milk then poured through a fine hair-sieve into a tub. All this was done under my eye. The smallest yield from any one cow at B farm was 1 Danish pound*, and the largest 10 pounds; the total weight of the milk, 119 pounds, which for 31 cows gives an average of 3·8 pounds of milk per cow. Mr. Estcourt, suggests that we should have run this quantity of 13 gallons of milk through a centrifugal separator, and apparently assumes the existence of a separator in every dairy. There was no separator at B farm, and Mr. Estcourt, with his exceptional experience, should be aware that some of the finest butter, both in England and Denmark, is made without a separator The leaving of the milk or cream for "ripening" is an essential of the Danish system of butter-making.

Mr. Estcourt considers that it was "dangerous" to leave the milk all night, even though it was sealed up. As a public analyst, I have had some experience of sealing, and knowing how the milk was sealed in the case in question, I am able to state positively that it was not tampered with. The seals placed on it by the British Vice-Consul (Mr. McGregor) and myself together rendered such a malpractice *absolutely impossible.*

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* 1 Danish pound = 500 grammes = 1·1 lbs. English.

Mr. Estcourt concedes that the supervision of the milking at the Danish farms "was performed by gentlemen whose honour, intelligence, and high ability in the special professions to which they are devoted cannot be questioned," but on the ground of their entire lack of experience of the new calling they undertook, Mr. Estcourt thinks "grave objection might well be taken." Mr. Estcourt is evidently unaware that two of the gentlemen referred to, Mr. Faber and Mr. Böggild, have gone through a course of training in dairy-farming amounting to a practical apprenticeship, and Mr. Böggild, in his capacity of consulting Dairy Chemist to the Royal Agricultural Society of Denmark, actually spends his life in visiting the Danish farms, and giving assistance and advice in the manufacture of dairy-products!

It is quite correct that the whole of the dairy operations were conducted under the unremitting supervision of the whole party, and I added the words, "especially Mr. McGregor and myself," because *we* checked the weighing, set our seals on the milk and the finished products, and took other precautions with the express purpose of preventing any imaginable fraud, and of meeting such criticisms as those of Mr. Estcourt.

It is a curious fact that while Mr. Estcourt suggests that the Danish farmers succeeded in surreptitiously introducing foreign fat under the very noses of the party, regardless of the consequences of their detection in the act, from various other sources I am hearing of samples of butter fully as abnormal in character as B and O. In fact, the more general cry is that our experience was not novel.

I sent Mr. Estcourt somewhat more than 30 grammes of the fat rendered from B butter, and am surprised to learn from his paper (page 56) that this quantity was found by him insufficient for the determination of the specific gravity by the Westphal balance.

It is a fact generally recognised that the result of the distillation of butter fat by Reichert's process, expressed as butyric acid, gives a somewhat lower figure than the soluble acids, expressed as butyric acid. But Mr. Estcourt found his G sample to require, when examined by Reichert's process, an amount of alkali corresponding to 4·16 of butyric acid, while the soluble acids in the same sample are stated as equivalent to 3·53 of butyric acid!

I will not ask Mr. Estcourt to state which of these two figures is erroneous, as I write this chiefly as a personal explanation, and do not intend to discuss the matter further.

QUANTITATIVE ESTIMATION OF ADULTERANTS IN LARD.

By H. W. Wiley.

The progress of qualitative analysis has rendered it possible for the skilled analyst at the present time to detect with certainty every commercial adulteration of lard. It is probably true that laboratory adulterations amounting to only 2 or 3 per cent. may escape the quest of the skilled chemist; but when adulterations are made for commercial purposes the amount of adulterant added is always in sufficient quantities to render its qualitative detection easy. For quantitative purposes, however, the matter is not so readily determined. For practical purposes the two adulterants which are used in making compound lards are cotton oil and the stearines derived by pressing partially crystallised lard or tallow. The first of these stearines is usually called prime lard stearine, and the second oleo-stearine. The following methods have been proposed for the quantitative detection of these adulterants.

1. *The amount of insoluble residue obtained upon treating the samples with a mixture of ether and alcohol.*—This method, as was shown in the celebrated trial of McGeoch, Everingham & Co., against Fowler Bros., in Chicago, was wholly unreliable, and it may be dismissed from the category of useful methods.

2. *The specific gravity.*—This method has great value, and may be relied upon to give approximate results.

3. *Absorption of iodine.*—This method would be an excellent one for determining the amount of cotton oil added to a lard, provided that no stearine was present; but the careful addition of cotton oil and stearine will enable the mixer to make a lard whose iodine number is almost identical with that of the pure article.

4. *The rise of temperature which samples undergo when mixed with sulphuric acid.*—This process, devised by Maumené, may give valuable information in regard to the quantity of adulterants added. Unfortunately, however, it is open to the same objection as lies against the iodine method, viz., the rise of temperature with the stearine is much less than that with lard, so that by the addition of stearine and cotton oil a mixture can be made in which the rise of temperature is not very much greater than that produced by lard itself.

5. *Melting point of the samples.*—This method is of but little practical benefit when taken alone. Mixtures of lard and cotton oil do not show a mean melting point as indicated by theory. Cotton oil melts one or two degrees below zero, while pure lard melts at about 40°. A mixture of equal quantities of these two substances shows a theoretical melting point of 20°. Such a mixture, however, will melt only a few degrees below the fusing point of pure lard.

6. *Refractive index.*—I am not aware that the refractive index has been used as a method of quantitatively approximating the amounts of cotton oil and stearine added, and I propose to say a few words in regard to its value in this respect. Fourteen samples of lard, known to be pure hog-grease, examined in my laboratory, gave the following mean data:—

Refractive index	1·4620
Percentage of iodine absorbed	62·48
Specific gravity at 35°	·9053
Rise of temperature with sulphuric acid	41·5°
Melting point	40·7°

Eleven samples of steam lards passed by the Chicago Board of Trade gave as a mean the following data:—

Refractive index	1·4623
Percentage of iodine absorbed	62·86
Rise of temperature with sulphuric acid	39·9°
Specific gravity at 35°	·9055
Melting point	37°

Thirteen samples of Armour's mixed lards gave the following data:—

Refractive index	1·4634
Percentage of iodine absorbed	63·58
Rise of temperature with sulphuric acid	46·5°
Specific gravity at 35°	·9060
Melting point	40·6°

Sixteen samples of Fairbank's mixed lards gave the following data :—

Refractive index	1·4651
Percentage of iodine absorbed	85·31
Rise of temperature with sulphuric acid	57·9°
Specific gravity at 35°	·9095
Melting point	38·1°

Sixteen samples of purified cotton oil gave the following data :—

Refractive index	1·4675
Percentage of iodine absorbed	106·84
Rise of temperature with sulphuric acid	83·7°
Specific gravity at 35°	·9145

Making use of these data, we reach the following results :—

Determined by the rise of temperature with sulphuric acid, Fairbank's lards con- ned 32·80 per cent. of cotton oil, and Armour's 11·39 per cent. Calculating from eir respective specific gravities, Fairbank's lard contained 45·65 per cent. of cotton oil, d Armour's 7·60 per cent. Determined by the iodine absorption alone, Fairbank's lard ntained 52·29 per cent. cotton oil, and Armour's 3·6 per cent. As determined by the fractive index, Fairbank's lard contained 56·36 per cent. cotton oil, and Armour's 25·45 r cent.* The unknown quantity, however, is the effect which the stearines employed d upon the data given. An oleo-stearine examined in my laboratory had an iodine sorption of 18 per cent.; a prime lard stearine an iodine absorption of 44 per cent. us more than twice as much of the oleo-stearine could be used as of the lard stearine thout perceptibly influencing the percentage of iodine absorption. The specific gravity the lard stearine used was about the same as that of the lard, viz., ·905; although a ger number of determinations might show a lower specific gravity.

The refractive index of one sample of oleo-stearine calculated at 25° was 1·4646. fore formulating any rule in regard to the matter I readily admit that a much larger mber of samples of stearine should be examined, and their refractive indices deter- ined. I think, however, it will appear finally that both the specific gravities of the arines employed, and the refractive indices will be found not to vary greatly from the mbers for pure lard; the specific gravities being somewhat lower. It is seen by the ove result that the approximate quantity of cotton oil in lard, as indicated by the fractive index, is much nearer the true proportion for the Fairbank and Armour mples than that given by any of the other methods employed. For instance, let us ppose that the Fairbank samples were composed of 60 per cent. cotton oil, 20 per cent. re lard, and 20 per cent. lard stearine; the quantity of iodine absorbed by such a lard uld be as follows :—

$$
\begin{aligned}
60 \times 107 &= 6420 \\
20 \times 62 &= 1240 \\
20 \times 44 &= 880 \\
100 \text{ parts} &= 8540
\end{aligned}
$$

Iodine absorption theoretical	85·40 per cent.
Actual iodine number obtained by analysis	85·31 "

Thus from the above theoretical calculation the amount of cotton oil added was) per cent., which as indicated by the refractive index was 56·36 per cent., by the ecific gravity 45·65 per cent., and by the rise of temperature with sulphuric acid ·80 per cent.

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* The above numbers are calculated from the mean data for the fourteen samples of pure lard d the sixteen samples of purified cotton oil.

In the case of Armour's lard take the following:—

	Ingredients, Per Cent.		Iodine Number.		Total.
Pure lard	70	×	62	=	4340
Cotton oil	20	×	107	=	2140
Oleo-stearine	10	×	18	=	180
	100			=	6660

Theoretical iodine number .. .. 66 60 per cent.
Actual iodine number .. .. .. 63·58 „

Let us suppose again that the compound was made with prime lard stearine, in which case we have the following computation:—

	Ingredients, Per Cent.		Iodine Number.		Total.
Pure lard	70	×	62	=	4340
Cotton oil	15	×	107	=	1605
Lard stearine	15	×	44	=	660
	100			=	6605

Theoretical iodine number .. .. 66·05 per cent.
Actual iodine number .. .. .. 63·58 „

From the above computations the value of the refractive index in determining approximately the respective quantities of cotton oil and stearine in mixed lards is apparent. It is true that in individual cases the variation might be very much greater than indicated above, but as an expression of the mean result it appears to me that the refractive index is fully as valuable if not more so than the specific gravity in the quantitative determination of mixed lards.

I propose to push this investigation somewhat further by more extensive examinations of the specific gravities and refractive indices of lard, oleo-stearine, prime steam lard, and mixed lards.

The refractive index of pure water at 25° as indicated by the instrument employed (Abbe's large model) was 1·3300. When the index of water at the above temperature is taken at 1·3330, ·0030 should be added to the numbers given in the above paper.

ON SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. Atwater.

(*Continued from page* 59.)

From these results, as well as from consideration of what is known of dissociation in general, it is evidently impossible to predicate anything definite regarding the amount of decomposition that would take place at a given temperature in a glass tube containing soda-lime and such a complex mixture of gases as occur in an ordinary combustion. I regret the lack of facilities which prevented determinations of the temperature at which the combustions in these experiments were made; but so far as one can judge from observation, I should say that what I have here designated as "medium heat" or "usual heat," namely, that in which the combustion tube is dull red and the soda-lime yellowish, is about that at which combustions are very commonly made in the laboratories where I am acquainted.

Prehn and Hornberger have reported observations which imply large losses of nitrogen by oxidation in soda-lime combustions. When sugar was burned in the tube to expel the air before the combustion and to sweep out the ammonia afterwards, considerably more ammonia was obtained than where this precaution was omitted, and the apparent oxidation by the air present in the tube at the beginning was greater than that from the air used in the aspiration at the end. But the experiments were made with ammonium salts, and where no sugar was used there was apparently no gas to drive out the air and prevent its oxygen from acting on the ammonia. In ordinary combustion of nitrogenous materials large quantities of gases are produced, and in the experiments I have just described oxalic acid was used, which naturally drove out the air.

On the other hand, numerous analysts find that with proper precautions there is no loss from oxidation. In our work we have taken pains to have the tubes well filled with soda-lime and substance so as to leave only very little open space for air, and to heat the anterior layer of soda-lime before applying the heat to the mixture of soda-lime and substance, thus expelling a large part of the air in advance, and have been careful not to aspirate with air until after putting out the flames, so that the contents of the tube have become somewhat cooled before the air has been admitted. In how far omission of these precautions may explain the apparent oxidation elsewhere observed, I, of course, cannot say. Naturally there would be the less danger of loss the more the air is removed by diluting gases before heating. But considerable experience has left me with the very strong impression that with the precautions I have suggested there need be very little fear of oxidation. This must be especially true where, as in ordinary combustions, large amounts of hydrocarbons are present to combine with the oxygen.

Makris has shown that oxidation may take place, but his experiments were made under conditions especially arranged to insure admixture of air with ammonia at a high temperature.*

Prehn and Hornberger have also experimented upon the effect of different degrees of heat on the ammonia in determinations with ammonium salts and potassium ferrocyanide. They find, at what they call ordinary heat or dull redness, no considerable indications of dissociation, but on heating to bright redness they found with tubes 35-40 cm. and the anterior layer of soda-lime 15 cm. long, considerable, and with tubes 55-65 cm. and the anterior layer 30-40 cm. still more indication of the dissociation of ammonia. Their results thus agree with those above given, except that they get more dissociation with the high heat in the long than in the short tubes. This they very reasonably explain by the fact that the ammonia in the long tubes had more time to dissociate, from which I infer that the gases were caused to pass more slowly than was the case in our work.

Makris also tested the dissociation of ammonia by passing a slow current of gas through a tube 70 cm. long filled with pieces of soda-lime and heated to bright redness. Analysis of the gases coming from the tube showed a very considerable amount of dissociation. But, as Gruber has observed, the case here was different from that in an ordinary combustion, in that a large amount of ammonia was exposed for a long time to a high heat and without any considerable amount of diluting gas, whereas in ordinary combustion there is less free space in the tube, and that largely filled with other gases, so that the ammonia is rapidly swept away, and furthermore the diluting gases are largely hydrocarbons which would naturally furnish nascent hydrogen to regenerate dissociated ammonia.

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* Ann. Chem. (Liebeg) 184, 376.

My results above detailed accord very exactly with those of Gruber and of Johnson and Jenkins above cited, who found no indications of loss by dissociation. But it is evident that both the complete transformation of nitrogen into ammonia, and the loss of ammonia, depend largely upon the manipulation. Thus Kissling gets very good results with an anterior layer 35 cm. long, and evident loss of nitrogen when the anterior layer is only from 7 to 10 cm. long, the combustion being carried on slowly.*

The experiments described by Mr. Ball and myself in a previous article bear upon the questions of dissociation of ammonia and of incomplete ammonification of nitrogen of distillation products. They were made with casein, the conditions being varied so as to allow in some cases very little, and in others considerable opportunity for the ammonia formed to be dissociated. The results, given in detail in the article referred to, are more concisely set forth in Table IV.

(*To be continued.*)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

ESTIMATION OF GLYCERIN IN THE COMMERCIAL ARTICLE. F. FILSINGER. *Zeitschr f. Angew Chemie, No.* 1, 1889.—The acetin process recommended by Benedict and Cantor (see ANALYST, 1888) gives good results with the purer kinds of glycerin, but is unreliable for the titration of samples got from soap leys, as these retain impurities which affect the process. Better results would no doubt be obtained if the impurities were first got rid of, but this would involve dilution, and the process only works with fairly concentrated samples. L. DE K.

TEST FOR ANTIFEBRIN IN PHENACETIN. M. J. SCHRÖDER. *Nederl. Tydschr. v Pharmacie,* etc. January, 1889.— Phenacetin, when taken internally, yields phenetidin and para amido phenol, both harmless bodies, whilst antifebrin yields aniline, which is decidedly poisonous. It is therefore of importance to test for the presence of antifebrin in phenacetin. The author found the best test to be Plugge's reagent, which consists of a solution of mercurous nitrate with a little nitrous acid. ·5 grm. of the sample is boiled in a test-tube with 8 c.c. of water, allowed to cool, and filtered off from the re-crystallized phenacetin. The filtrate is boiled with a little potassium nitrite and dilute nitric acid, then mixed with some of Plugge's reagent, and again boiled. If no red colour is got, the sample may be considered as practically pure; at all events, there cannot be more than 2 per cent. of antifebrin.
L. DE K.

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* Ztschr. anal. Chem. 24, 1885, 448.

ZINC SALICYLATE. L. VAN ITALLIE. *Nederl. Tydschr. v. Pharmacie*, etc ebruary, 1889.—The author tests the salt by incinerating the compound, moistening e ash with nitric acid, and finally igniting the residue, which should be not less than per cent. A commercial sample only yielded 18 per cent. The author estimated the gree of solubility of this salt in various fluids. One part dissolves in 25·2 parts of ater at 16° C. One part of the anhydrous salt dissolves in 36 parts of ether of 725 . gr. at 16° C., and in 450 parts of chloroform of 1·495 sp. gr. at 15° C. One part the salt dissolves in 3·5 parts of spirits of wine, sp. gr. 819·4 at 15° C. In petroleum irit it is quite insoluble. L. DE K.

MODIFICATION OF KJELDAHL'S NITROGEN PROCESS. J. W. GUNNING. *Nederl. ydschr. v. Pharmacie*, etc., February, 1889.—The author operates as follows: One part of tassium sulphate is fused with two parts of sulphuric acid. This mixture gets semi-lid in the cold, but it readily melts, and may then be treated like a fluid. About one amme of the substance to be analysed is put into a 300 c.c. flask with round bottom d short neck. If liquids such as milk or beer have to be tested, a suitable quantity ust first be evaporated to dryness in the flask itself. About 30 c.c. of the acid mixture e now added, and the whole heated with a Bunsen burner. At first strong frothing curs and white fumes escape, consisting chiefly of water vapour. To prevent loss of rong acid, the neck of the flask is now fitted with a funnel, which is then covered with watch-glass. This simple arrangement will now cause the acid to condense and run ck into the flask. The operation is finished when the acid looks colourless, which will generally the case after about an hour. After cooling, the ammonia is now estimated usual. The author prefers the standardising of the volumetric acid by the iodine and dium hyposulphite method. The test analyses are very satisfactory. Uric acid elded 33·3 per cent. of nitrogen, theory requiring 33·33 per cent. Aniline oxalate elded 10 per cent., theory requiring 10·1 per cent. L. DE K.

ASSAY OF CARBOLIC ACID. L. DE KONINGH. *Zeitschr f. angew Chemie, No.* 5.—he author, in reply to Mr. Williams, still believes the salt test to be a good one, both the liquefied acid of the B.P. and hydrated cresylic acid. If the acid contains much r oil, it cannot be in the hydrated state, but a little uncombined water may be shown the benzol test, which, however, is not meant to be used for the purer forms of the id. The percentage of tar oils is best estimated by agitating the sample with four mes its volume of 10 per cent. soda ley if necessary, with addition of a fixed quantity benzol. Williams' process for the estimation of free acid in carbolic powders is no ubt a most excellent one, but the author fails to see in what essential particular it ffers from the one published in the ANALYST, vol. xii., by Dr. Muter and himself. To intents and purposes it is just the same, though perhaps not quite so accurate, cause not performed in specially constructed apparatus. W. H. D.

STANDARDISING PERMANGANATE. R. JAHODE. *Zeitschr f. angew Chemie, No.* 4, 89. -Many analysts still prefer to standardise their permanganate with pianoforte wire, but e trouble always is to exclude the air after the iron has dissolved. The author operates follows: The iron is dissolved in boiling acid in a flask, which is closed by a cork, through which goes a doubly-bent tube, the end of which is made to dip into a beaker containing a solution of sodium bicarbonate. When solution is complete and the liquid allowed to cool, the soda solution finds its way into the flask, but no sooner have a few drops got in, than an evolution of carbonic acid gas setting in drives the fluid back.

L. de K.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the Analyst.

Sir,—In the number of the Analyst for February last, there is a report of a paper by Mr. Rowland Williams on the "Determination of Citric Acid in Lemon Juice," brought before the Society of Public Analysts, "to promote," as Mr. Williams said, "a discussion among the members." I should have been glad to have taken part in such a discussion, had there been one, if Mr. Williams had, in pursuance of his object, kindly informed me that his paper was to be read, and that the greater part of it had reference to what he interpreted as being my method of "Estimating Citric Acid in Concentrated Lemon Juice," and in condemning the use of a solution of an alkaline carbonate for the titration. If Mr. Williams had informed me some months ago, when he asked for some information, that my very general reply was to form the foundation of a paper to be read before the Society, I might have described to him the precautions necessary to be observed in sufficient detail to have saved him from being misled into the belief that there is any objection to the use of alkaline carbonate for the purpose, or of running the risk of misleading others, beyond a little more time being required for the experiment than is necessary when caustic alkali is employed. There is absolutely no inaccuracy involved in use of alkaline carbonate, with litmus as an indicator, when the proper conditions are appreciated and observed.

I have no kind of intention of taking up the space of the journal with a criticism of Mr. Williams' paper as a whole, but I cannot help expressing my surprise at the figures given in the two tables which accompany it.

In the first of these are given comparative results of the analyses of six samples by the caustic method and by alkaline carbonate, showing, according to Mr. Williams, that the carbonate gives uniformly and exactly one ounce of citric acid more in a gallon than the caustic solution.

The only possible explanation of there being difference at all, is that there was interference by carbonic acid retained in the solution; but if Mr. Williams allowed carbonic acid to remain during the final testing, how is it there is anything like agreement in the results and that the differences are almost constant?

In the second table the results of the analyses of seven samples of pure citric acid by the two solutions are given, and the same astonishingly uniform differences are shown.

I cannot think Mr. Williams means to suggest that the alkali in an alkaline carbonate has less saturating power for citric acid than it has as it exists in the caustic condition, and yet this seems to be the only possible inference to be drawn from the tables themselves. My explanation, of course, is that these differences are really errors of manipulation.

I do not think I should have taken up the space of the journal with this letter if I not lately found that Mr. Williams' paper has been distributed widely in pamphlet form amongst manufacturers and dealers commercially interested in the sale of lemon juice, of whom some, at all events, are not in a position to estimate correctly the value of Mr. Williams' criticism of what he believes to be my method of analysis, but which, in fact, is only his mode of employing it. At the meeting at which the paper was read, there seems to have been no discussion, Mr. Allen only expressing what appears to me, to say the least of it, a very hasty "regret" that "chemists of repute" should adopt an alkaline carbonate with litmus as an indicator for the purpose under discussion. Now, in reply to Mr. Williams and to Mr. Allen, I should have thought it might have been assumed by the Society of Public Analysts that chemists of repute, who had been engaged in a special analysis for five-and-twenty years, had carefully examined and tested every process that had been suggested, and under their sense of responsibility, and as the result of their long experience, had adopted the process, which in their hands gave the most reliable returns under all circumstances. Many hundreds of analyses have been made in my laboratory with caustic and with alkaline carbonate solution side by side, and observing the precautions necessary in each case, I, unlike Mr. Williams, obtain identical results, and I protest as strongly as possible against the inferences to be drawn from Mr. Williams' tables, as being opposed to all my experience. There are reasons why sodium or potassium carbonates are frequently to be preferred. After removal of the carbonic acid with properly prepared litmus paper of the proper tint, there is absolutely no objection to their use. As a matter of fact, I do not think Mr. Williams finds it easy to obtain caustic alkali free from carbonate.—I am, sir, yours truly,

39, Lime Street, London, E.C., G. H. Upton.
26th March, 1889.

THE ANALYST.

MAY, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A GENERAL meeting of the Society was held on the 10th ult. at Burlington House.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened, it was announced that the following gentleman had been elected as a member :—Mr. W. J. Dibdin, F.I.C., F.C.S., London.

The PRESIDENT said that it was only fitting he should refer to the recent death of Mons. Chevreul, who was an hon. member of their Society. Some years ago he had sent them a letter wishing the Society every success, and he was sure they would all regret his loss. Mr. Allen also made some remarks in allusion to the same subject.

The following papers were read and discussed :—

"Contributions to the Study of the Action of Water on Lead Pipes," by A. H. Allen.

"Iodine Absorption of Butter Fat," by Rowland Williams.

"'Pure' Chemicals," by Bertram Blount.

"Some Experiments on the Soap Test," by Dr. E. Waller.

The next meeting of the Society will be held at Burlington House on Wednesday the 8th May inst.

NOTES ON LACTOSE.

BY E. W. T. JONES, F.I.C.

(*Read at the Meeting, March* 1889.)

ABOUT twelve months ago I carefully purified some milk-sugar, and subsequently obtained some very nice crystals, which after draining, etc., were finally dried in vacuo over H_2SO_4. The following notes of the results obtained with them I have every confidence in publishing, and although I do not propose to give all the experiments in detail, the results may be taken as very carefully fixed by repeated experiments.

5 grms. (exactly) of the crystals (=4·75 grms. anhydrous lactose) were put into a correct 100 c.c. flask, about 80 c.c. distilled water added, and solution effected by heating

in boiling water for twenty minutes to half an hour. The next day the flask was filled up at 60° F. to exactly 100 c.c. Thus I avoided bi-rotation.

The sp. gr. at 60° F. of such a solution is 1018·6, hence at this concentration the divisor for sp. gr. above 1,000 to get grms. per 100 c.c. of

$C_{12}H_{22}O_{11}$ is 3·91
and $C_{12}H_{22}O_{11} + OH_2$ is 3·72

Using a Soleil-Ventzke-Scheibler polariscope, I find at this concentration the sp. rot. pr. for

$C_{12}H_{22}O_{11} = 60{\cdot}5[\alpha]_j = 54{\cdot}6[\alpha]_D$
$C_{12}H_{22}O_{11} + OH_2 = 57{\cdot}5$ „ $= 51{\cdot}9$ „

Cupric reducing power with Fehling solution, using not less than 30 c.c. (diluted with two volumes of water) for 0·1 grm. lactose, heated in vessel surrounded by *boiling* water for fifteen minutes (O'Sullivan's method).

$CuO \times 0{\cdot}5723 = C_{12}H_{22}O_{11}$
$CuO \times 0{\cdot}6024 = C_{12}H_{22}O_{11} + OH_2$

I weigh as CuO, *i.e.*, I convert the Cu_2O into CuO by ignition in a porcelain crucible —the whole secret of sure complete oxidation being to use *at first* a *very small* flame to *just char* and destroy the filter paper, after which more heat may be applied and final ignition over the blow-pipe resorted to—proceeding thus the CuO *does not cohere*, and there is not the slightest risk about not obtaining full oxidation by ignition alone—duplicates should not, and generally do not, in my hands, differ more than a milligram.

Action of citric acid.—I have corroborated that lactose is not affected optically, nor in cupric reducing power, by this acid, when proceeding as follows, whilst cane-sugar is completely inverted under such circumstances. Crystallised citric acid is added at the rate of 1·6 grms. per 100 c.c. of solution. Generally I take 50 c.c. of sugar solution and add 0·8 grm. of citric acid, and heat in 100 c.c. flask in boiling water for thirty minutes, and then make up to within half a grm. of the original weight. During the heating a little more loss in volume occurs by evaporation than the increase caused by the citric acid. The weight of the flask, solution and acid being taken before heating, and afterwards made with distilled water to within 1 grm. for 1·6 grm. citric acid, or within half a gramme for 0·8 grm. citric acid, exactly the same volume is obtained after treatment with acid as before, and, therefore, if only lactose is present, the polariscopic readings will be the same.

The proportion of citric acid recommended increases the bulk of the solution $\frac{1}{100}$th, viz., 1·6 grms. citric acid to 100 c.c. makes it 101 c.c., and 0·8 grm. to 50 makes it 50·5 c.c., hence we arrive at the proper allowance for adjusting the solution to the original volume.

I have said that cane sugar is completely inverted, and may I be pardoned for mentioning here that although the real sp. rot. pr. of invert sugar is, $23{\cdot}75[\alpha]_j$, *i.e.*, calculated on the increased molecule of the original cane-sugar, if calculated on the cane-sugar, as generally will be the case in practical work, 25·0 must be used.

Crystals of lactose do not lose their water of crystallisation by twenty-four hours' heating in a water oven, but if the crystals be just dissolved in water and evaporated over a water bath to dryness, and then dried in the water oven for a few hours, anhydrous ctose is obtained; but the drying must be continued till the weight is constant, which roceeds slowly if the residue is thick. It has been stated that our residues containing actose after drying in the water oven contain this body in the hydrated state, but this dispute, if drying is conducted till constant weight occurs.

2·3810 grms. of the crystals after drying for twenty-four hours in the water oven, weighed 2·3806, practically no loss.

0·6828 grm. crystals just dissolved by addition of distilled water, then evaporated to dryness, after drying four or five hours in the water oven, became constant at 0·6500 grm. against 0·6487, the correct amount of anhydrous lactose corresponding to the crystals taken.

It is quite needless for me to point out the practical application of these results in such analyses as those of condensed milk, etc., etc.

COLOURING MATTER (ARSENICAL) USED FOR COLOURING CANDLES.

By W. F. Lowe, F.I.C., F.C.S., Associate of the Royal School of Mines.

(*Read at the Meeting, March*, 1889.)

Whilst the use of dangerous mineral colours for colouring tinned and bottled fruits, wall papers, etc., has been pretty nearly stamped out, their use for colouring wax candles appears to have been overlooked. The danger of using them for such a purpose has recently been brought to my notice, and a brief description may be of interest to the members of our Society.

A children's party was given by a gentleman in the district for which I am Public Analyst, and a Christmas-tree with a large number of small coloured candles was the principal attraction. On the day following the party about twenty per cent. of the children who had been present, and also several grown-up persons, suffered from symptoms of irritant poisoning. At first it was naturally supposed that something in the food had caused the mischief; but on making inquiries the gentleman found that some of those attacked had taken nothing to eat or drink in the house, but had been present at the Christmas-tree, so that it could not possibly be the food. His suspicions then fell on the green candles which had been used for lighting the tree, and as he had had a chemical training, he examined one of the green candles himself, and found traces of arsenic in it. He then forwarded the remainder to me for examination, and I found the green ones contained both copper and arsenic, evidently having been coloured with one of the arsenical greens, which appeared to be "emerald green."

There was not sufficient material to estimate with any accuracy the amount present, but I was able to separate a small quantity of the colouring matter itself by dissolving the wax in warm ether. It is well to note that at first it appears as if the colouring matter had also dissolved, for it is in such a finely divided state that it floats about in the ether for some time, thus giving the appearance of a green solution; but if it is filtered allowed to stand a few hours it is seen that it is merely in suspension.

From information I have obtained, it appears to be a common practice e smaller candle works to use arsenical greens for colouring candles, as they are easier use than the aniline green, and stand the action of light so much better, the candles coloured with aniline greens having a tendency to fade unless kept in a dark place.

The quantity employed, I am informed, is from ½ oz. to over 1 oz. of emerald green to 28 lbs. of wax, and that for the small green candles about 1 oz. to 28 lbs. would be employed. At this rate each small candle, I find, would contain about ¼ grain of colouring matter, and would be capable of yielding about $\frac{1}{8}$ gr. of arsenious anhydride; so that I should say that a dozen or two of such candles burnt in a room of moderate size would be capable of producing very serious effects.

Red candles also are usually coloured with an injurious mineral colour, at least for such a purpose, for vermilion is used, and a red candle from the same Christmas-tree, I find contains vermilion, but I believe the candles used were chiefly green ones. I intend to make a further examination of the matter, and I hope some of the other members will do the same, for I think that such a practice should, if possible be stopped.

DISCUSSION.

Mr. **HEHNER** said that about twelve years ago he was just about to publish a note on the same subject when he found that Prof. Church had already drawn attention to the matter.

Mr. **CASSAL** said that the fact of arsenic having been found in candles was not new. Some years ago the question had been brought forward, and it was alleged that some people had been actually poisoned by arsenical candles, but if he recollected rightly they were *white* candles, not coloured ones. The subject was of very considerable interest and importance from a hygienic point of view. The statements that the practice was being stamped out he could not agree with. Most analysts still had a good many things to examine for the presence of arsenic. Some years ago, at the time of the Health Exhibition, he had occasion to examine a number of articles for arsenical and other poisonous pigments, among them butterfly nets, coloured balls, and various children's toys, arsenic in large amount being frequently found. The green gauze used for nets had been found to contain arsenic.

The point was the extreme minuteness of the quantity by which the injury may come about. It would be a good case to help Mr. Dyer, as the whole chain of evidence was complete.

With regard to the use of arsenious acid in candles, he understood it was used to give the burning wick the turn; and if that were so, he was surprised some evil effect had not been discovered.

As to the rash mentioned, that was quite a fact; he had heard of several instances of rash being produced by arsenical pigments, which have an irritating effect on the skin.

The whole matter was very interesting to him from a medical as well as chemical point of view, with the experience he had had.

Mr. **ALLEN** said that the paper just read showed the desirability of extending the Sale of Food and Drugs Act. Public analysts ought to have under their care all such articles as arsenical wall-papers, fabrics of candles, besides disinfecting powders, &c. The subject was not strictly a novel one, but it was only by calling attention to cases which came within their own experience that it was brought home to them, and he thought they were indebted to the author for bringing the matter forward. He might remind them of the historical case of a member of the royal family of Austria who was practised on by introducing arsenic into the candles used in his bedroom. It was not fair to judge of the poisonous effects of arsenical paper or candles from the amount of arsenic present, for the finely-divided arsenious oxide would be so distributed through the atmosphere as to affect the system far more strongly than if an equal amount had been taken into the stomach; in fact, the effects would probably be more properly omparable with those attending the breathing of arseniuretted hydrogen, which was nown to be intensely poisonous.

He should like to know what became of the copper in the candles. How any andle could contain a measurable or weighable amount of copper which did not go into the wick he could not understand; in fact, if he had had to analyse such a candle, he thought he should have burned the candle and condensed the arsenic by some contrivance similar to that for estimating sulphur in coal gas, and should have looked in the candle end for the whole of the copper which had been previously distributed throughout the candle.

Mr. BERNARD DYER said he would be glad if Mr. Harvey would send him the medical details of the case. It might be known to members that this question of injurious colouring matters, not only applied as to papers, but to other things, had been taken up at various times by various societies, but more especially by the National Health Society. The Society of Arts had previously collected evidence and made a report, but nothing came of it. The National Health Society, in 1883, appointed a committee consisting of medical men and chemists to draft a Bill for the suppression of the sale of arsenical wall paper, on the same lines as the Acts of Parliament in operation on the Continent and, he believed, also in America. In Germany, he believed, arsenical paper was made and exported to this country, but it could not be sold in Germany. In Sweden there was a very stringent law on the subject. The great difficulty in drafting a Bill was in defining the word "arsenical." After considering the question for some time, Mr. Carr, Mr. Heisch, and Dr. Bartlett suggested a very excellent modification of the Marsh apparatus which seemed to meet all difficulties, both as to testing and defining arsenical colours. Dr. Willoughby was at first secretary of the committee, and was succeeded by himself (Mr. Dyer), and the Bill became finally matured during his (Mr. Dyer's) secretaryship, in 1885; and Dr. Cameron undertook to bring the matter before the House of Commons. But the Irish question came up, and Dr. Cameron said it would be perfectly hopeless to bring this forward then, as it would certainly be opposed by the large trade interests, and as he, moreover, was very busy; so nothing was done, and the draft Bill was still on the shelf awaiting an introducer and an opportunity of introduction. The fact remained that England is almost the only civilised country where arsenic-dyed wall papers were allowed to be sold.

One of the great difficulties Dr. Cameron said they would have to contend with was that, as large trade interests were involved, and there would be much opposition, a Select Committee would be doubtless appointed, and that Committee would ask them for their medical evidence. Now, the Society of Arts and the National Health Society had gathered a drawer full of medical evidence, but much of it related to cases where the arsenic was so abundant that, on wiping the paper with a duster, the green came off on the cloth, and much more to cases where patients being out of health, and wall paper being found arsenical, the two facts were assumed to be cause and effect on grounds which, though very strong, did not afford strictly accurate and convincing evidence.

They felt that, before the matter could be brought forward with full confidence, they should strengthen their medical evidence. Any strictly authentic cases of domestic poisoning from arsenic he should be glad to receive on behalf of the National Health Society, that he might hand them to Mr. Kenneth Millican, who had undertaken the collection and collation of medical facts on the question.

Mr. CASSAL said that general practitioners among medical men who had not studied one were not likely to give specially valuable opinions on the points referred to—ast, that was his experience, which was not a small one. Mr. Cassal mentioned the case of a lady who had been wearing a bonnet with bronzed leaves in it, some of these leaves having been kept in close contact with the skin. The leaves contained a large quantity of arsenic, and the lady had suffered from an obstinate skin eruption and other arsenical symptoms. Another lady who had bought some coloured "Indian" muslin at a large establishment in the West End, and worked at it with her maid, had noticed that both of them were beginning to suffer from inflammation of the eyelids. The muslin was brought to him for analysis, and he found large quantities of arsenic. The vendor had said that he would eat all the arsenic that could be found in it, and he (Mr. Cassal) was sorry he did not do so. The sulphide was at his service.

In another case five or six children were being kept in a room with a large fire in it, the walls being covered with a green arsenical paper. The symptoms had been mistaken for those of a violent common cold.

Dr. Muter said he had had some similar Indian muslin sent to him, and he also found arsenic in it.

Dr. Harvey stated that he frequently met with arsenical wall papers. Only on the previous day a case was brought to his notice of serious illness traced to a drab-coloured bedroom paper, which upon analysis was found to contain a considerable amount of arsenic. He was acquainted with numerous other cases where injury appeared to have resulted from the use of such papers.

Mr. Adams said that more than twenty times he had had patients suffering from arsenical poisoning. It was a common result that the eyelids became affected; they had a peculiar red appearance which a practical oculist at once knew to be due to arsenic. He had a case quite lately showing what a very minute amount would do the mischief. Of course, in the case of the candles it must have been a very small quantity that each person could have breathed. The poisonous effects of arsenic depended in a measure on two things—first, the idiosyncracies of the person (some could take it with impunity whilst others are injuriously affected by the smallest amount), and secondly, if it gets into the system by the air cells of the lungs it is absorbed much more quickly and injuriously. A lady patient of his had some fur which he had examined and found to contain arsenic. She wrote to him about it on the previous day as follows:—"I am very pleased to tell you anything I can about the fur you found contained arsenic. Directly I began to wear it I had every symptom of a very bad cold in the head, and later on this was accompanied by sore throat and diarrhœa. Naturally in hot rooms I always felt worse." The lady put the fur on one side and at once recovered. When she resumed wearing it, all the symptoms recommenced. This experience was repeated again and again till she came to associate the symptoms with the wearing of the fur, which was then sent to him, and he found out the cause.

THE METHODS FOR DETERMINING FAT IN MILK.

By Dr. P. Vieth, F.C.S., F.I.C.

(*Read at the Meeting, March,* 1889.)

On looking over the chemical literature of the last decade, we find that much attention has, during that period, been paid to the determination of fat in milk. So numerous are the methods brought out, and the alterations and improvements suggested, that it seems to me, that the supply far exceeds the demand, and that it becomes necessary to classify the various methods in divisions and subdivisions, when attempting to briefly review them in anything like a lucid manner. I will with your permission try to do so in the remarks which I am going to make.

I. Methods which can be worked outside the chemical laboratory, and by persons having no analytical training.

1. Processes confined to the dairy.

(*a*) *Control apparatus* worked in connection with the Danish Centrifugal Cream Separator. No chemicals required. The milk samples are simply subjected to centrifugal force in suitable vessels, and the volume of cream read off. This volume indicates the percentage of fat sufficiently near for all practical purposes.

(*b*) *Lactocrite*, worked in connection with Swedish Cream Separator. The milk is treated with strong acids, whereby the fat is, so to speak, set free, and under the influence of centrifugal force unites, so that its volume can be determined. It is generally admitted that results thus obtained agree very closely indeed with those arrived at by determining the fat gravimetrically. At the same time it is rather curious that some experimenters, *e.g.*, W. Blyth, compare it with Adams' method, while others—among them, Soxhlet—compare it with the latter's method; both parties find agreeing results, and still paper and plaster method do not give results agreeing among themselves. It should be mentioned that a smaller machine has been constructed, which can be used in the laboratory of the analyst, although, I am afraid, the price will not facilitate its introduction.

2. *Optical Methods*.—They are based upon the assumption that the opacity of milk stands in direct relation to the amount of fat the milk contains. Not only is this supposition wrong, but the test is also influenced by a number of uncontrollable circumstances. The results are, therefore, always doubtful, frequently quite fallacious. Feser's Lactoscope is, relatively speaking, the best of all the optical tests.

3. *Soxhlet's Areometric Process*.—Milk is mixed with potash solution, and then shaken with ether. The ethereal fat solution is allowed to separate, and its specific gravity determined. A table gives the corresponding percentage of fat. The results may be considered to be identical with those arrived at by extracting milk dried up on plaster of Paris.

4. *Cronander* and *Liebmann* shake a mixture of milk and potash solution with ether. After complete separation they allow the ether to evaporate and measure the volume of the fat left behind. I do not think these methods recommend themselves, and cannot find that they have been taken up.

5 *Marchand's Lactobutyrometer*.—Milk is shaken in a gauged tube with ether to dissolve the fat; shaking is continued after the addition of alcohol, and the tube then placed in warm water. The mixture separates into three layers; the volume of the upper one, containing the fat, is read off. Every one-tenth c.c. indicates ·2 per cent. of fat, in addition to 1·2 per cent. which is kept in solution. The results are fairly correct in the majority of cases, when dealing with milk which is not too rich.

6. *Short* heats milk with strong alkali in a boiling water-bath for two hours, decomposes the soap by the addition of a mixture of sulphuric and acetic acid, allows the liberated fatty acids to rise, and determines their volume, which is then enlarged in the proportion of 87 to 100. This method seems to be rather troublesome, and open to grave objections. The results are said to be lower than those obtained by Adams' method.

II. Methods which must be considered as confined to the chemical laboratory, and the hands of the analyst.

7. *Extracting the fat* from the residue left when milk is evaporated without the addition of an inert substance.

(*a*) No care is taken to disintegrate the residue (Wanklyn).

(*b*) Pains are taken that the residue should be in a readily exhaustible condition (Dr. Jas. Bell, Carter Bell).

The difficulty of completely exhausting such milk residues, more especially if the milk is poor in fat, is now generally recognised and the methods may be looked at as abandoned.

8. *Muter* proposes to precipitate the casein which carries down the fat with it; to wash the precipitate first with water, then with alcohol, and at last with ether, which is collected and evaporated, when the fat is left behind.

9. Milk is dried up on some inert substance and then extracted. The substances suggested are glass powder, sea sand, plaster of Paris, asbestos, pumice stone, filter and blotting paper, wood fibre, sponge; and these various modifications are connected with the names of Abraham, Adams, Babcock, Gantter, Johnstone, Macfarlane, Soxhlet, Storch, and others. This group certainly contains the most exact methods for the determination of fat in milk.

10. *Morse* and others suggest to dry milk on anhydrous sulphate of copper, extract the fat by means of light petroleum, saponify, and determine the quantity of alkali required for saponification. I find only one account of experimenting with this method, in which it is said that the results fall below those arrived at by using Adams' method.

11. *Roese* shakes milk first with alkali, then with a mixture of ether and light petroleum, reads off the volume of the upper layer after complete separation, and determines in an aliquot part of it the fat gravimetrically. An allowance is made for fat retained in the aqueous layer.

12. *W. Schmid* first heats milk with strong hydrochloric acid, after cooling shakes with ether, notes the volume of ethereal layer, and ascertains the fat in an aliquot part. Determining the fat in an aliquot part of its solution appears to me very undesirable, considering that one has to deal with ether, which is not only highly volatile, but the volume of which is also much influenced by temperature; besides, experimental errors are much increased.

This is a rather long list of methods for the determination of fat in milk, and still, I am convinced, it is very incomplete, containing only those methods which have come under my notice and impressed themselves on my memory. Of some of the methods enumerated there exist modifications with regard to the chemicals and apparatus employed.

Very generally acknowledged as standard methods are Adams' paper and Soxhlet's plaster processes; the indications of other methods which do not give direct results are compared with and gauged by either of these processes. Methods like those of Roese and Schmidt can claim no other advantage but speed combined with a near approach to correctness. Speed, no doubt, is a valuable item, and approaching correctly results may suffice under certain circumstances, but certainly not with regard to the official work of a Public Analyst. He ought to employ the most exact methods which are at his disposal.

As an apology for the deficiency of new matter in the foregoing remarks I may say, that they were originally not intended to be read as a separate paper, but meant to be thrown into a discussion which, however, had to be abandoned for want of time.

DISCUSSION.

MR. DYER suggested that calcined gypsum might give erroneous results by causing slight saponification of the fat, for gypsum usually contained some carbonate of lime which became caustic on calcination, and might make a lime soap.

DR. MUTER noticed that Dr. Vieth had referred to the method of coagulating the milk with acetic acid, collecting the curd, washing the same first with water and then with spirit, and finally percolating with ether. This was in certain cases a very good way of working as he had had occasion to before mention to the society.

MR. HEHNER said it would be very interesting to get a full catalogue of the different methods. As Dr. Vieth had gone so far perhaps he would go farther and complete it.

DR. VOELCKER corroborated Mr. Dyer, and said that only last week he had such a sample of gypsum sent to him.

Mr. Allen said that a good many years ago the late Mr. W. W. Stoddart, of Bristol, exhibited what was known as Horsley's process and tube, at a meeting of the British Pharmaceutical Conference, and also described a modified method by which the test was said to be applicable to butter. He, Mr. Allen, was very much interested in the description given, and consequently read a paper on the same subject at the following meeting of the conference, in which he expressed a very unfavourable opinion respecting the method. As a matter of fact, the process did not originate with Horsley, but had been described by Marchand many years previously. The whole subject had gone so completely out of his mind, that when a question was asked him in the "English Mechanic," as to the nature of the lacto-butgrometer, which was simply a Marchand's or Horsley's tube, he said he had never heard of the instrument. Dr. Vieth thereupon pointed out that the instrument had been advertised on the front page of the ANALYST, for a whole year previously, which he was afraid showed that advertisements were apt to be wasted on some people.

DR. VIETH in reply said, that he considered plaster of Paris when employed in milk analysis, an inert substance inasmuch as its desired action was a purely mechanical one. Plaster of Paris was alkaline, but they must remember that milk was not neutral, but displayed besides alkaline, also acid reaction. A number of experiments had shown him that the acidity of milk, was in excess of the alkalinity of the plaster of Paris which he used. He had no difficulty in getting good plaster from Hopkin and Williams.

With regard to the method of extracting the caseine, he had followed that himself in a great many instances, extracting the precipitate after it had been dried in the air bath; he found the results agreed very well indeed with the plaster extraction.

The Marchand's process gave very fair results in the majority of cases, of course, the results were not exact enough for the work of public analysts; but, for instance, in the hand of a farmer who wanted to pick out his best and worst cows, it gave very good results indeed. He had made thousands of determinations; when the fat was within the limits of 3 per cent. and 3·5 per cent. the results agreed very closely. When dealing with richer milks, the results were not so reliable. He would never recommend the process for the use of public analysts, especially at the present day, when the amount of fat could be calculated with great accuracy, from the figures for specific gravity and total solids.

(*Conclusion of the Society's Proceedings.*)

ON SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. Atwater.

(Continued from page 76.)

TABLE IV.

Effects of long heating and of open space in tube upon amounts of nitrogen obtained as ammonia from casein containing 12·43 *per cent. of nitrogen.*

Conditions of Experiments.	Nitrogen Lost.					
	In per cent. of Weight of Casein.			In per cent. of Total Nitrogen.		
	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
	per ct.	per ct.	per ct.	per ct.	per ct.	per ct.
No channel, long heating (2½ hours) ..	0·47	0·16	0·35	3·8	1·3	2·8
Channel, ¾ hour	1·92	0·05	0·65	15·4	0·4	5·2
Channel, long heating	2·95	0·00	0·68	23·7	0·0	5·5
Channel, long heating, high heat ..	..	..	8·51	..	..	68·5
Channel, long heating, long tube ..	9·88	9·29	9·58	79·5	74·7	77·0

When the tubes were closely packed so as to insure the maximum of contact between the gases produced and the heated soda-lime (water-vapour at high temperature), and the combustion was conducted at a moderate heat and kept within the usual time of about three quarters of an hour, the full amount of nitrogen, 12·43 per cent. of the water-free casein, was obtained as ammonia. But when the combustion proceeded very slowly so as to occupy two and a half hours, the other conditions remaining the same, only from 12·27 to 12·08 per cent. of nitrogen were obtained as ammonia, making a loss of from 0·16 to 0·47 per cent. reckoned on the weight of the water-free casein, or from 1·3 to 3·8 per cent. of the total nitrogen. It seems reasonable to ascribe this loss to dissociation during the long time that the ammonia was exposed to the heat, though possibly some of it may have been due to incomplete ammonification of nitrogenous decomposition products. It is easy to understand how the ammonia passing rapidly through the interstices between the particles of soda-lime in the closely packed tube, in the ordinary analyses, would be so mixed with water-vapour and other gases that the nitrogenous distillation products would be completely changed to ammonia, and in the very brief exposure to heat the resultant dissociation would be too small to be noticeable. On the other hand, when the operation extends through two and a half hours instead of three quarters of an hour, it would seem by no means improbable that the anterior layer of soda-lime might, before the end of this long period during which it is kept hot, cease to give off any considerable amount of water-vapour. But it is probably this water-vapour which yields the hydrogen to form ammonia with the nitrogen of the volatile distillation products, and it is not impossible that the same vapour may also tend by its presence to prevent dissociation of ammonia. Add the fact that, when the operation goes on slowly, the time of sojourn of the ammonia in the heated tube is of course relatively much longer than the increased time of combustion would imply, and a large loss by dissociation and incomplete ammonification is easy to understand.

When a channel was left in the tube, so that the contact between distillation products and the soda-lime was less intimate, the loss of nitrogen was still greater. When, with the channel, the combustion occupied only three quarters of an hour, this loss varied from 0·4 per cent. to 15·4 per cent. of the total nitrogen. But when the combustion, with the channel, was prolonged to two and a half hours, the loss reached in one case 27 per cent. of the total nitrogen. This increased loss would seem most probably due to both incomplete ammonification and dissociation. At any rate, the conditions provided for less perfect contact between soda-lime and substance; for reduction of the supply of moisture which would provide hydrogen for ammonification and would also tend to prevent dissociation; and for a very long exposure of ammonia to heat.

When, with channel and long heating as before, the temperature of combustion was raised as high as the tubes would endure, the loss was very much greater, and amounted to 68·5 per cent. of the total nitrogen. The most natural explanation of this increased loss is the increased dissociation of ammonia. When the length of the anterior layer of soda-lime was increased, and with it, consequently, the amount of open space inside the tube, the loss of nitrogen was greater still, namely, from 75 to 79 per cent. of the whole.

It would, of course, be foolish to attempt to say how much of the loss in these latter cases was due to dissociation and how much to incomplete ammonification. Such questions can be answered only by more detailed experiments, in which the gases produced should themselves be analysed. But it does seem reasonably safe to say that both dissociation and incomplete ammonification must account for a large part, if not all, of the errors here observed, and that there is great danger of loss in these ways unless due precautions are observed.

To conclude: As regards loss by dissociation and oxidation, the facts above cited appear to warrant the inferences that, in combustions conducted by the method here described, in which (1) the tubes were so closely packed with soda-lime as to leave a minimum of free space inside, (2) full opportunity was provided for contact between distillation products and soda-lime (water-vapour at high temperature) by the anterior layer of soda-lime 12 cm. long or thereabouts, which was well heated before applying the heat to the mixture of soda-lime and substance, and maintained at a medium heat during the whole operation, and (3) the operation was completed in not over three quarters of an hour:

1. Provided the tube and contents are allowed to cool slightly before aspirating with air, there need be no loss by oxidation.

2. At a temperature sufficient to heat the tube only to dull redness, there need be no considerable loss by dissociation.

3. Ammonia may be dissociated and nitrogen lost by either very high heat, or by conducting the operation so slowly as to leave the ammonia exposed for a long time to heat. Very likely the danger of long exposure is heightened by lack of moisture from the anterior layer of soda-lime after the latter has been heated for some time.

4. A vacant space in the tube (channel as ordinarily recommended) may involve serious loss. This loss is probably due to both incomplete ammonification of distillation products and to dissociation of ammonia.

The danger of loss of ammonia from rapid flow of the gases through the acid solution in the bulb-tube is sometimes assumed to be considerable. Musso, for instance, lays great stress upon uniform and slow evolution of gas, and prolongs the combustion for several hours, the main purpose seeming to be to insure complete absorption of the ammonia.* The experiments cited by Mr. Haynes in a previous article imply that the real danger from this source is very small. All his attempts to push the combustion fast enough to force ammonia through the solution were ineffectual. For instance, in a combustion of sulphate of ammonia, completed in 12 minutes, the ammonia, which was sufficient to neuteralise half or a little over half of the acid, was entirely absorbed. The volume of the acid solution was 10 c.c.; it was contained partly in the bottom bulb and partly in the outer bulb of an ordinary Knop and Arendt bulb apparatus, in accordance with our usual practice.

Bearing of the Results upon the Interpretation of those obtained by other Experimenters. The Difficulties with the Soda-lime Method observed by Seegen and Nowack, Musso, and others.

Of the investigations which have been interpreted as casting doubt upon the reliability of the soda-lime method, those of Seegen and Nowack have, perhaps, been the most frequently quoted. These have carried great weight because the figures obtained were so wide of the truth despite the evident pains taken to secure accurate results, and because similarly bad results have been obtained by other chemists. The apparently unavoidable sources of serious error in the soda-lime method have been alleged as a ground for doubting the correctness of important series of researches in which the soda-lime method has been used. The question of the validity of this doubt is a very important one.

In his account of his own experiments with flesh, Nowack states that care was taken to insure uniform development of gas, and the tubes were kept glowing hot for three hours ("die Röhre stets durch 3 Stunden glühen"). In the report of their joint experiments with animal and vegetable protein compounds, albumin, casein, syntonin, flesh, gluten, legumin, Seegen and Nowack state that they employed the same analytical methods and precautions ("die Analysen wurden in derselben Weise und midt denselben Cautelen ausgeführt, die . . . ausführlich erörtert sind") as were employed by Nowack in his own investigations just referred to. The inference is that their combustions were likewise continued through a long time, if not at a high temperature. Musso, whose experiments were made with milk, whey, and cheese, says his combustions lasted hardly less than three hours, and in some cases they were prolonged for even six hours. In the work of Nowack, of Nowack and Seegen, and of Musso, the figures obtained for nitrogen were very small. Taking those for nitrogen obtained by the absolute method as the standard, the loss of nitrogen in Nowack's experiments reached 20 per cent., and even more, of the whole nitrogen; in those of Seegen and Nowack the loss was from 1 to 20 per cent., while in those of Musso it was over 30 per cent. of the whole nitrogen.

On the other hand, Gruber, who in reply to the criticism of Seegen and Nowack upon analyses by Voit and others in Munich, gives very satisfactory results obtained in Voit's laboratory with protein in flesh, never allowed more than half an hour for the combustions.

The experiments cited by Mr. Woods and myself in article No. IV. of this series seem to us to give satisfactory assurance of the reliability of soda-lime determinations of nitrogen in ordinary protein compounds when the combustions were made with proper precautions. Of the latter, the chief seem to us to be (1) sufficiently fine pulverisation and careful mixture of the substance with soda-lime; (2) close packing of the tube and sufficiently long anterior layer of soda-lime, so as to insure sufficient contact between soda-lime (heated water vapour) and distillation products, as well as to avoid too long sojourn of ammonia in the heated tube; and (3) maintaining the anterior layer at a moderate heat during the whole time of combustion, which should not be too protracted.

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* Ztschr. anal. Chem. 16, 414.

Messrs. Seegen and Nowack do not state whether they left a channel in their tubes or not; but it is evident that the conditions of their work were such as to favour incomplete decomposition of the nitrogenous substance, imperfect ammonification of volatile decomposition products, and dissociation of ammonia. Prof. Seegen reports a nitrogenous residue in the tube after the combustions, and Dr. Nowack found the acid solution in the nitrogen bulbs so highly coloured as to interfere with titration with litmus solution. The combustion was prolonged for three hours, and during part of this time at a very high heat. The determinations of Muss were conducted very slowly, in some cases even more so than those of Seegen and Nowack, and the results obtained were likewise very low. Mr. Ball and I found that either leaving a channel in the tube, or high heating, or prolonging the combustion to two and a half hours, involved large loss of nitrogen, the loss with these conditions combined amounting, in some cases, to two thirds or three fourths of the whole nitrogen.

It would seem, therefore, that we have here cases of a kind not unfrequent in scientific research, in which the very effort to secure correct and reliable results involves grievous error.

I lay especial stress upon this matter because of the use that has been made of Seegen and Nowack's results to discredit the soda-lime method in general, and particularly to throw doubt upon the work of Voit and others in the Physiological Laboratory at Munich, in which this method has been employed in the study of the fundamental problems of nutrition. It was my fortune some time since, while working in that laboratory, to be permitted to observe very closely the ways in which determinations of nitrogen by soda-lime are there conducted. Although the effort to insure close packing of the tubes was perhaps less than in the experiments above detailed, and upon which the conclusions here given are based, yet on the whole the details of manipulation as I observed them were such as would, with the experience in this laboratory, lead one to expect correct results.

Although the results of experiments by Gruber and others in the Munich laboratory, and accumulated experience elsewhere, make any vindication of its work superfluous, yet I trust these statements, in connection with an explanation of the error into which Seegen and Nowack have unwittingly fallen, may, in the interests of science, not seem out of place.

The experience of other chemists, as well as that in this laboratory, of which part has been described in these pages, leaves me with the decided belief that the Scylla and Charybdis of the soda-lime determination are incomplete ammonification of nitrogenous distillation products and dissociation of ammonia. The other difficulties are, in general, easy to overcome. They may all be prevented, in ordinary animal and vegetable protein compounds, by the precautions above enumerated. But in alkaloids and allied compounds, and in some amines, and amido and azo-compounds, loss by incomplete ammonification seems very difficult to avoid.

The observations I have had occasion to make, as well as printed accounts of the methods followed in different laboratories, have given me the decided impression that the most common difficulties are loose packing of the combustion tube so as to leave a channel, insufficient anterior layer of soda-lime, and too high or too long-continued heating.

The soda-lime method is beset with dangers, and requires great care to avoid them. Indeed, after the experience in this laboratory, covering a period of several years and including some thousands of nitrogen determinations, if I were going to undertake again a series of analyses like those for which these studies were made, and the details of the work were to be placed in the hands of an analyst, however expert, who had not been through some such experience as that above described, I should feel under the necessity of asking him to do a considerable amount of preliminary work, including comparison of results by the soda-lime with other methods, before I could feel sure of his results. A case in point may be worth mentioning.

After most of the above detailed experiments had been made and we had the method in such control that we were wont to say jocosely that from a given protein compound we could obtain the whole or any desired fraction of the nitrogen as ammonia, at will, a gentleman of no little experience, and who afterwards proved himself a skilful and efficient analyst, came to our laboratory as assistant. We placed a number of substances, mostly animal tissues, in his hands for nitrogen determinations by the soda-lime method, instructing him in the details of the process, and going through a number of determinations with him. He was very confident that he could secure perfectly reliable results and at the same time make the determinations with considerable rapidity. After a list of substances had been analysed, I looked over his figures, and was somewhat surprised at the variations in the duplicate determinations, which, in some cases, as I now recall, reached nearly 0·2 per cent. This led me to question their accuracy. Mr. Woods, by whom most of the nitrogen determinations of this investigation have been made, repeated the analyses and found in almost every case a considerably higher percentage of nitrogen, so that the whole work had to be repeated. The most plausible explanation of the errors seemed to be that the determinations in question were made rather hurriedly, and either because the nitrogenous material was not well enough mixed with the soda-lime, or from too loose packing of the tube, some of the nitrogenous distillation products were incompletely ammonified, and thus escaped detection in the titration subsequent to combustion.

At the same time I must repeat what I have already said, that our experience leads me to place the greatest confidence in the soda-lime method for the determination of nitrogen in ordinary protein compounds, provided the work be conducted with the precautions here insisted upon.

The perfection to which Kjeldahl's method has lately been brought, and its accuracy, convenience, and inexpensiveness, have led to its use in this as in many other laboratories. Our experience leads us to decidedly prefer it to the soda-lime method, though we find it advantageous to use both, making one check the other. But the danger of incomplete ammonification of some classes of compounds, *e.g.*, alkaloids, makes us feel it necessary to control both by the absolute method for all classes of substances, except those for which they have been most thoroughly tested.

SUMMARY.

The experimental and other considerations presented in this and the previous articles on the determination of nitrogen by soda-lime, may be conveniently summarised, after first recalling the probable reaction by which the nitrogen is changed to ammonia, and the principal sources of error in the operation.

1. It seems decidedly probable that the change of nitrogen to ammonia is effected by union, at high temperature, with water vapour yielded by the soda-lime (or slaked lime in case the latter is used). It is essential that the contact between nitrogenous distillation products and water vapour be sufficient, and not at too high or too low temperature to insure conversion of all the nitrogen to ammonia, and that the ammonia be not dissociated or oxidised. The main objects, then, are to secure complete ammonification and to avoid dissociation and oxidation.

2. The chief difficulty in the way of complete ammonification of protein compounds appears to be the formation of gases which do not readily yield their nitrogen to be united with the hydrogen of the water vapour. With certain other classes of nitrogenous compounds, as leucine and its congeners, alkaloids, amines, and amido and azo-compounds,

is difficulty is greater, and sometimes apparently insuperable. The tendency of protein mpounds to be decomposed by heat and other agencies into leucine, amines, etc., appears explain the difficulty frequently found in getting all their nitrogen into the form of nmonia by heating with soda-lime. The evident means to secure complete ammonifica- n must be sufficient contact with soda-lime at proper temperature.

3. The danger of dissociation of ammonia evidently increases with increase of tem- rature and time of exposure, and is probably diminished by presence of water vapour d other diluting gases. If this be so, the danger will be avoided by measurably rapid mbustion at not too high heat, and by keeping the ammonia in contact with sufficient oisture from the soda-lime until it leaves the heated tube.

4. Leaving out of account substances such as nitrates, nitro-compounds, etc., whose trogen is imperfectly converted into ammonia by soda-lime, even in the presence of organic atter, and assuming palpable errors of manipulation, to be avoided, such as (*a*) loose pack- g of asbestos plug, which would allow particles of soda-lime to be swept into the acid bulb;) heat at anterior end of the tube, so low as to permit ammonia to be retained with oisture about the cork, or so high as to char the cork and give rise to acid or alkaline stillation products; (*c*) use of soda-lime containing nitrates or nitrites, which may, ac- rding to circumstances, either furnish nitrogen to be transformed into ammonia, or ygen to burn the ammonia formed from the nitrogen of the substance; (*d*) use of dis- lled water containing ammonia for rinsing the acid bulb; (*e*) imperfectly-cleaned or correctly-calibrated burettes; the principal sources of error above discussed involve ss of nitrogen, and may be recapitulated thus :

I. Loss from imperfect ammonification of the nitrogenous substance, due to :—

a. Incomplete decomposition of the substance, part of the nitrogen being, from arseness of the particles of the substance, imperfect mixing with the soda-lime, insuffi- ent heat, or other cause, left behind in the charred residue.

b. Change of nitrogen into compounds other than ammonia, either such as may main in the tube, *e.g.*, cyanogen; or volatile distillation products which escape ammo- fication and pass through the acid solution unabsorbed, or, if absorbed, are not accu- tely determined by the titration or other means used to find the amount of nitrogen the solution.

c. Escape of nitrogen in the free state.

II. Loss of ammonia through :—

a. Dissociation at high heat in the combustion tube.

b. Oxidation by air present in the tube before, or introduced in aspirating to wash t ammonia after, the combustion.

c. Neutralisation by acid products, *e.g.*, of sugar, where the latter is used in the mbustion.

d. Incomplete absorption by the acid solution.

5. Complete decomposition of the substance has, in our experience, been readily cured by pulverisation fine enough for it to pass through a of seive of 1 mm. aperture; orough mixing with soda-lime; avoiding the shaking by which the particles gather at e top of the soda-lime; and heating to low redness.

6. With sufficient soda-lime, not too dry, we have found no reason to fear the for- ation of cyanides, nor have we been able to obtain any indication of the escape of free itrogen when the operation is properly conducted, although it might, perhaps, occur by xidation of ammonia if there were nitrites or nitrates present, or if aspiration with air ere done while the tube and contents are hot. Turning off the flame before aspirating as, in our experience, sufficed to avoid oxidation by air. At least, if ammonia has been xidised, the quantity has been too small to be detected.

7. When sugar is used, acid products may be formed in quantities large enough to impair the accuracy of the determinations. With ordinary animal and vegetable protein compounds, provided enough soda-lime is employed, the use of hydrogen or sugar, or other substances for supplying gases, either to expel air, or to yield nascent hydrogen to form ammonia with the nitrogen, or to dilute the ammonia and prevent dissociation, or to wash out the ammonia, appears to be unnecessary. The danger of loss by incomplete absorption of the ammonia by the acid solution appears to be very small indeed, even when the development of gases is very rapid, provided sufficient acid solution be used.

(*To be continued.*)

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

ABNORMAL BUTTERS: A REPLY TO MR. ESTCOURT'S PAPER IN "THE ANALYST," MARCH, 1889.

SIR,—Mr. Estcourt appears to have had two objects in view in his paper on "Abnormal Butter," viz.: (1) to show that Mr. Allen failed to prove the existence of genuine butter of an abnormal composition, and (2) to defend the conclusion drawn from his analysis of a sample of butter from Wigan.

As to the first point, Mr. Allen has already answered. So many cases of abnormal butters have been found of late, that had Mr. Allen not brought the subject before the Society, some other analyst would surely have done it by this time.

On the second point, I may explain why at the time I considered the question of the Wigan butter to be one of abnormal butter, but shall at once admit that—as far as Mr. Estcourt is concerned—I was mistaken. Had Mr. Estcourt been more willing to discuss the matter at an earlier stage, the whole case would probably have received quite a different treatment.

Some butter was sampled at Wigan last summer and pronounced to be adulterated. Mr. Estcourt, who analysed the sample for the defendant, certified it to be "a sample of margarine," "having regard both to its specific gravity and the very low per centage of soluble fatty acids." This is the sample No. 402 of Mr. Estcourt's paper.

Some samples of butter were taken at the dairy from which this butter No. 402 was believed to have come. These were marked "G" and "N."

Some of these samples G and N were sent to Mr. Estcourt, who wrote of them, "The specific gravity of both is lower than that of any genuine butter I have yet analysed. The soluble fatty acids are also not present in such a per centage as to indicate a genuine butter." From the figures in his paper on "Abnormal Butters," it appears that he found No. 402, G and N, almost identical in their composition.

Professor Stein found those samples, G and N, to yield about 24 c.c. by the Reichert-Wollny process, and a small sample of butter-fat sent him from the agent of the shipper of the Wigan butter, who had obtained it from Mr. Estcourt as the remainder of sample 402, yielded the same amount of volatile acid, viz., 24 c c.

The two analysts were therefore agreed upon the very close similarity between these three samples. I shall not enter into any discussion as to the supposed genuine nature of sample G; it is not to the point here. I merely wish to deny ever to have written about "the supervision of the milking" from which samples G and N were obtained. I only note that both analysts found the Wigan butter and samples G and N very similar; of G and N samples exist still in the original packing and with unbroken seals.

The genuine nature of a butter-fat yielding 24° c.c. by R.-W. may well be questioned, as such butter-fat is of rare occurrence, which fact has made Professor Stein name it "abnormal." He did at first question the genuineness of the butter from the dairy in question, but having learnt the history of sample G he—rightly or wrongly—altered his opinion, and pronounced this butter genuine but abnormal, and having found the sample of butter-fat, said to be the remainder of Estcourt's sample 402, to be very similar to sample G, he concluded that abnormal butter of this composition was not known to Mr. Estcourt.* Having at the time no knowledge of the actual results of Mr. Estcourt's analyses, I concurred in Professor Stein's view, and I concluded that a demonstration of the existence of genuine butter of this composition, taken with sufficient guarantee, would suffice to show that the analysis of the Wigan butter (402) was not sufficient proof of its adulteration. I do not yet see how we could have come to a different conclusion.

My first step was then to write to Mr. Estcourt (on Sept. 18th), asking him for the particulars of his analyses of the samples, mentioning that the Wigan case was "likely to throw some new light on the question of the variations of volatile or soluble acids in butter." Mr. Estcourt replied (Sept. 20th) that he should "be pleased to exchange particulars of analyses" with me, if I would give him the results obtained by the chemist in Denmark. On Sept. 28th I called on Mr. Estcourt in Manchester, and gave him the figures he has published, as obtained by Professor Stein, being all that Professor Stein had at that time estimated in the three samples. I naturally understood that Mr. Estcourt would give me his figures in return; but although I urged him to do so, he declined, promising, however, that he would send them on three days later. *I have never since heard from him.* It is somewhat surprising that he should complain that he "heard no more of this matter," for I left him with the understanding that I was first to hear something from him.

My plan to discuss with Mr. Estcourt the question of the existence of genuine butter yielding 24 c.c. R.-W., had, of course, to be given up after this breach of promise. I therefore sought and obtained the assistance of Mr. Allen.

Had Mr. Estcourt, according to his promise, made both by letter and verbally, informed me that he had found about 3·5 per cent. of soluble fatty acids in these samples, I should then have seen at once that the question was not one of "abnormal butter," but of "abnormal analysis," and I should have proceeded accordingly. It could never enter my mind that a sample of fat containing only 3·5 per cent. of soluble acids could be genuine butter-fat; but neither could I ever be expected to suppose that in a sample yielding about 24 c.c. by R.-W., anybody could find only 3·5 per cent. of soluble acids.

Mr. Estcourt speaks of "the Reichart method." He does not mean that, however. From the way in which he uses the name "Reichart," I gather that he means the Reichert-Wollny method, which is described by Mr. Allen as the one used by him and Professor Stein. Mr. Estcourt argues against this method, that it is liable to give erroneous results, in extreme cases—according to Wollny—30 per cent. *too low.* But he does not mention, although he quotes the very paper by Wollny, that these errors are those of the original process, and that they are impossible when using the method as modified by Wollny! But, even should Mr. Estcourt have employed the original and faulty Reichert's method, how did he succeed in getting a result which is just 30 per cent. *too high?*

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* As stated previously, Professor Stein had met with abnormal butter yielding even less than 24 c.c. the year before this Wigan case.

" I desire to call attention to the paper by Dr. Wollny in the January and February numbers of THE ANALYST for 1888," and I ask, how is it possible that an analyst should find 30 per cent. *too high* a result, even if he cares to ignore the improvements to the method? Wollny finds that carbonic acid in the original process may cause an error of + 10 per cent. *as a maximum.*

After the publication of Mr. Estcourt's paper, I asked Professor Stein to estimate the soluble fatty acids in samples G and N, and in the remainder of the small sample obtained from Mr. Estcourt as No. 402. With his permission, I publish the results of his analyses, obtained by the modification of the method suggested by Dr. Muter. I give (1) the per cent. of soluble acid; (2) the result by Reichert Wollny; (3) the per cent. of butyric acid corresponding to the number of c.c. of $\frac{N}{10}$ found by R.-W.; and finally (4) the per cent. of butyric acid found in this way by R.-W., expressed as per cent. of the total amount estimated by Muter's method. To this I add the results of Mr. Allen's analyses of samples B and O, and of Mr. Estcourt's analyses of samples 402, G, N, B, and O, all calculated in the same manner.

	p. c. Soluble Acid.	R.-W. c.c.=	p. c. Butyric Acid=	p. c. of Total Soluble Acids.
		BY PROF. STEIN.		
402	4·7	24·0	4·22	89·8
G	4.70	24·2	4·26	90·6
N	4·79	23·9	4·21	87·9
		BY ALLEN.		
B	4·44	22·39	3·94	88·7
O	4·68	24·70	4·35	92·9
		BY ESTCOURT.		
402	3·40	..	..	..
G	3·53	23·60	4·15	117·6
N	3·57	22·55	3·97	111·2
B	4·51	22·44	3·95	87·6
O	4·70	24·64	4·34	92·3

The only acid in butter-fat which is volatile, and not soluble, is capric acid, but as it amounts to less than one per cent. of the total amount of soluble acids, it is of no consequence. As only about four-fifths of the solution is distilled off in the Reichert-Wollny process, only part of the volatile acids pass over in the distillate, viz., about 90 per cent. Mr. Estcourt, however, has succeeded in distilling over considerably more than the total amount of the soluble acids! In sample G he found 3·53 per cent. of soluble acids, 90 per cent. hereof is 3·177, which calculated to 5 grams. of fat, and expressed as c.c. of $\frac{N}{10}$ solution of butyric acid is 18. This figure, therefore, should be the result by R.-W. of a sample of butter containing 3·53 per cent. of soluble acids. Mr. Estcourt found 23·60, or, he found either 30 *per cent. too high* a result by Reichert-Wollny (which beats the record), or he found 1 *per cent. too low* a result by estimating soluble acids, which means that *over* 20 *per cent. of the soluble acid escaped his notice.*

As his opinion of the Wigan butter is based on his estimation of the soluble acid, it is therefore not surprising that he should consider it adulterated. I must however necessarily doubt the correctness of his analysis, and thereby his right to conclude, that the Wigan butter was adulterated. Yours faithfully, Harald Faber.

To the Editor of the Analyst.

Sir,—I shall only occupy a very small space in reply to Mr. Allen's reply.

I desire to call especial attention to the statement which he boldly makes, (apparently from his own knowledge), that I for years used a Westphal balance of which the plummet was broken, and the results obtained with it which were incapable of being published.

In answer to this, I can only say I am pleased to believe that the most of Mr. Allen's statements have a better foundation on fact than this, or the scientific world would suffer.

Mr. Allen does not give his authority for this incorrect statement, so he must have evolved it from his inner consciousness.

Since 1876, when I first introduced the Westphal balance to chemists in this and other countries, for ascertaining the specific gravity of liquids at high temperatures, I have always had in my possesion at least one duplicate bulb made by Westphal, for my instrument, which is also subdivided by Westphal to indicate half degrees.

In November, 1885, I suggested to Mr. Allen that he should give up quoting specific gravities, at 100 F. and use my apparatus and bath, &c.

He thanked me for my suggestion and altered the whole of the gravities in his new volume in accordance with the method which I have been using since 1876.

This much of ancient history I have given in justice to myself, as it will serve probably better than anything else to show the improbability of Mr. Allen's statement.

With regard to Mr. Allen's other statements, they are already sufficiently answered in my paper published in the March number of the Analyst,

The allusion, in the last two paragraphs, to the Reichert result of G sample serve to show what has been already stated, that the Reichert process gives erratic results.—I am, sir, yours,

Charles Estcourt.

To the Editor of the Analyst.

Sir,—In connection with the articles on Abnormal Butter which have appeared in recent numbers of the Analyst, the following lines may be of interest.

In December, 1885, both at the beginning and the end of the month, I made in my Laboratory butter from the milk of a single cow, and this butter, both in my own hands and those of Mr. Shippen Wallace, Chemist, Philadelphia, Pennsylvania, gave by the Reichert test figures ranging from 11·3 to 11·5 c.c. of deci-normal alkali consumed in neutralizing the first acid distillate of 50 c.c. I found the insoluble fatty acids (by Hehner's process) to be 89·6 per cent.

As stated by Wallace and myself (*Ninth Annual Report*, N.J. State Board of Health, 1885), the cow was one of a herd of eight cows in Princeton, N.J. She was of mixed breed, chiefly Alderney; nearly five years old; had a calf about eight months before, and was expected to have another late in the following spring; yielded six to seven quarts of milk daily at that time; her food was barley meal and corn-stalks (maize); she was apparently in sound condition. The butter made separately from five other cows in the same herd, within the same week or nearly so, ranged from 12·2 to 15·1 c.c. deci-normal alkali, so that neither the season of the year nor the food appeared to have any influence on the result.

In consequence of this result, we recommended that the minimum for butters made from the milk of a single cow should not be above 11 c.c. of deci-normal alkali.

I have no doubt that the milk was delivered to me in its normal condition, because I sent my own private messenger to obtain it; the owner of the herd did not know what use was to be made of it; exactly the same result was obtained from the milk at intervals of a month (or within very narrow limits, the same results); and finally, the milk from five other cows in the same herd was normal as to the behaviour of the butter fat.

While this is not intended to have any especial bearing on Mr. Estcourt's paper on Abnormal Butters, Analyst, March, 1889, it is presented as evidence that pure butter fat from the milk of a single cow, under normal conditions so far as we can judge, may yield by Reichert's method, figures decidedly below the usual standard.

I would add that I place more dependence on Reichert's than on any other process, and do not hesitate to condemn by its results butters which I can know have been made in this country and from the mixed milk of several cows; accepting then as high a standard as 11·5 to 12 c.c. of deci-normal alkali.—Respectfully yours,

H. B. Cornwall.

John C. Green School of Science,
Princeton, New Jersey, U.S.A.
March 20th, 1889.

To the Editor of the ANALYST.

"ON THE DETERMINATION OF CITRIC ACID IN LEMON JUICE."

SIR,—Will you kindly permit me to make a brief reply to the letter of Mr. G. H. Ogston (not *Upton*) on the above subject, which appeared on page 86 of the April number of the ANALYST?

Mr. Ogston says he would have been glad to have taken part in the discussion on my paper had there been one, and complains of my not having informed him of my intention to criticise the method which he employs for the examination of lemon juice. This was surely quite unnecessary. Mr. Ogston is a member of the Society of Public Analysts, and would no doubt receive due notice of the title of my paper, so he could presumably have attended the meeting if he desired. I can scarcely be held responsible for Mr. Ogston's absence, and I can assure him that no one anticipated and wished for his presence more than myself. Mr. Ogston is quite mistaken when he insinuates that, some months previously, I wrote asking him for certain information with the object of making it the basis of my paper. My sole reason for communicating with Mr. Ogston was because there had been complaints from calico-printers in this district at the discrepancies which were sometimes found to occur between my figures and Mr. Ogston's, and I thought it would be to our mutual advantage if our results agreed more closely in future. The explanation offered by Mr. Ogston with regard to the cause of the difference between our results, viz., the presence of carbonic acid in my titrations is, though plausible enough, altogether inadequate. I am quite certain that no carbonic acid could have been present, as in all the experiments every possible precaution was taken to ensure accuracy.

I followed Mr. Ogston's own directions as carefully as possible, viz., "Have the test paper nearly neutral and light in colour. After the addition of about two thirds of the necessary quantity of soda solution, boil for ten minutes to expel carbonic acid, and boil between each subsequent addition of the test." There seems to be some uncertainty in Mr. Ogston's mind as to whether I meant to suggest that the alkali in sodium carbonate has a different saturating power for citric acid than the alkali in caustic soda. What I clearly meant, and what I still contend is that, when titrating solutions of citric acid with sodium (or with caustic soda for that matter), using litmus paper as indicator, the point of saturation is reached sooner (owing to the alkaline reaction of sodium citrate to litmus paper) than when caustic soda and phenolphthalein are employed, consequently the sodium carbonate appears stronger than is theoretically possible. For instance, suppose 7 grains of pure citric acid are dissolved in water, and the solution titrated with normal caustic soda, using phenolphthalein as indicator, 100 fluid grains will be required. But if, on the other hand, normal carbonate of soda solution and litmus paper are employed, the addition of, say 97 grains, with subsequent thorough boiling and cooling, will in all probability be sufficient to produce a distinct blue colour; 100 grains of the alkaline solution would, therefore, appear capable of neutralizing 7·2 grains of citric acid, owing to the unsatisfactory nature of the indicator employed. For obvious reasons Mr. Ogston seems to be aggrieved because reprints of my paper have been distributed among those interested in the matter, but I may say that the majority of these persons entirely approve of my action.

While writing, I have before me a letter from the chemist at a well-known calico-printing works, confirming my statement with regard to the greater accuracy of citric acid determinations made with caustic soda and phenolphthalein.

For my own part, I know of no "reasons why sodium and potassium carbonates are frequently to be preferred," as is stated by Mr. Ogston near the close of his letter.

Mr. Ogston may protest as strongly as he pleases against the inferences to be drawn from my tables, but, to slightly alter the words used by Mr. Allen in the discussion which followed my paper, and which Mr. Ogston rather strangely ignores, "there can be no doubt on which side the truth lies.
—I remain, sir, yours faithfully, ROWLAND WILLIAMS.

28, Pall Mall, Manchester,
April 10th, 1889.

CATALOGUE of Chemical and Physical Apparatus, and Chemicals Manufactured and Imported by Philip Harris and Co., Limited, Bull Ring, Birmingham. This is a very complete and exceedingly well got up catalogue, the illustrations being numerous and well executed. The list of physical apparatus is most complete, and does great credit to the firm.

A TREATISE on Manures; or, the Philosophy of Manuring. A practical handbook for the Agriculturalist, Manufacturer and Student, by A. B. Griffiths, Ph.D., F.R.S. (Edin.), F.C.S. London; Whittaker and Co.

ERRATUM. Mr. G. H. Ogston, F.C.S., F.I.C., 39, Lime Street, E.C., was the writer of the letter, which, by a printer's unfortunate error, appeared over the signature, "G. H. Upton," in our last issue.

THE ANALYST.

JUNE, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A GENERAL meeting was held at Burlington House on the 8th inst., the President, Mr Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

Mr. A. J. Hailes, Analytical Chemist, Hornsey, was proposed as a member.

The PRESIDENT said that the Council would be glad to receive from members before the next meeting (this month) suggestions as to where the usual country meeting should be held; one or two places had been mentioned Liverpool and Paris—but before finally deciding, the Council would like to know the views of members on the question.

The following papers were read and discussed:—

"On the discrimination of the various species of Saccharomycetes," by Dr. W. J. Sykes.

"Fat Extraction from Milk Solids," by H. D. Richmond.

The papers announced by Mr. Faber and Dr. Vieth were, owing to the lateness of the hour, postponed until the next meeting.

In the course of the evening the President, at the suggestion of Mr. Cassal, invited Dr. Voelcker to inform the meeting of the result of his contest with the Bedford Town Council.

Dr. Voelcker accordingly referred to the dispute he had had with the Bedford authorities. He said he had no wish to bring before the Society any matter of his own, and had simply taken up the question as one which concerned all public analysts. A full report of the case will be found on another page, but briefly the circumstances were these: In his capacity of public analyst for the borough of Bedford, Dr. Voelcker on one occasion had fourteen samples sent to him to analyse. Of these, thirteen were perfectly good, and one, a sample of coffee, was found to be adulterated with 80 per cent. of chicory. In returning the certificate of this adulterated sample, and of the others, he did not put in the weights of the samples. As a matter of fact, the weights had all been taken, although they were not mentioned on the certificates. On this ground the Town Council *of Bedford, after* several meetings, decided to refuse payment of the fees for any of these fourteen analyses. The adulterated sample, they first said, they would make the basis of a prosecution, and after applying to him (Dr. Voelcker) to attend on a certain day, they wrote subsequently that they were sorry no prosecution could be instituted, because the certificate was informal. He offered to send an amended one; but the clerk to the Board, who happened to be a solicitor, said he was of opinion that after the certificate had once passed into his hands it could not be altered, so the prosecution would have to be abandoned. In the end the Town Council passed a resolution that none of the fourteen analyses should be paid for.

He (Dr. Voelcker) reported this to the Council of the Society of Public Analysts, who considered the decision absurd, and hoped he would put himself in communication with Mr. F. Low, the Hon. Solicitor to the Society, and fight the matter out.

He accordingly gave the matter into Mr. Low's hands, and on April 11th attended at Bedford with Mr. Low for the trial of the case in the County Court. It came before his Honour Judge Bagshawe, who, after an hour's discussion, reserved his decision, after expressing himself strongly of opinion that the plaintiff (that is, Dr. Voelcker) had no case at all. The report states that the Judge was satisfied that the certificate must be made out according to the statute, and after it had once left the analyst's hands he could not alter it. The Council's solicitor contended that the plaintiff had left out a material part by not inserting the weight, and the analysis was not complete until a proper certificate had been given. A further contention was, that they were entitled to a certificate for every sample, whether genuine or not, and that a letter to say a sample was genuine would not be sufficient; that they had entered into an arrangement with him, and until he had handed them a certificate legally made out, the work was not complete, and should not be paid for.

Although he (Dr. Voelcker) offered to be present so that no certificate would have been requisite at all, the Town Council said, "No; we are entitled to have a certificate which would enable us to dispense with your attendance and proceed without you."

Mr. Low requested the Judge to consider the question as to whether the certificate could not be amended, and the matter was then adjourned.

At the end of April he (Dr. Voelcker) received from Mr. Low a copy of the following letter, which had been forwarded to him from Bedford:—"Voelcker *v.* Mayor, etc., of Bedford. The judge has given judgment for the plaintiff for £19 19s., including the £6 6s. paid into Court; the costs are to follow the event, and payment is to be made in a month." This was a deduction of one guinea from the total amount of the claim. The costs were to be on the £20 scale. He was not able to say what the deduction of one guinea was for, whether for one certificate that was stated in Court not to be signed (though this was not stated in the pleadings), or for the one upon which it was alleged no prosecution could be instituted.

The PRESIDENT congratulated Dr. Voelcker on the result of the action, and thanked him for the public spirit with which he had acted in the matter, which was of interest to all public analysts.

On the proposal of Mr. CASSAL, seconded by Mr. STOKES, it was unanimously resolved that the thanks of the Society be tendered to Dr. Voelcker for the public spirit he had exhibited in carrying the matter to a court of law.

The **President** said he thought also they had good reason to be well satisfied with the way in which their Hon. Solicitor, Mr. Low, had acted in the matter.

At the next meeting of the Society the following, amongst other papers, will be read :—

"Condensed Milk," by H. Faber.

"On the quantity of Volatile Acids present in Butter Fat," by Dr. P. Vieth.

ON THE IODINE ABSORPTION OF BUTTER FAT.

By Rowland Williams, F.I.C., F.C.S.

(*Read at the Meeting, April,* 1889.)

Previous to Mr. Allen reading his valuable paper on "Some Abnormal Samples of Butter" before the Society of Public Analysts in December last (Analyst, vol. xiv., p. 5) I had on a few occasions determined the proportion of iodine absorbed by the filtered fat of various butters submitted to me for analysis.

Since then I have applied the iodine absorption test to a large number of butters and butter substitutes, in the hope of obtaining information of a nature reliable enough to be of service in certain cases where the methods usually employed in the examination of butter have failed to give the analyst complete satisfaction.

It has, I think, been pretty well established by Mr. Allen and others that butter of a perfectly genuine character, but possessing somewhat abnormal properties, might be condemned as sophisticated, if, for instance, the analyst based his opinion too implicitly on the proportion of volatile and soluble fatty acids present in the sample. A knowledge of this fact has latterly placed chemists in an awkward predicament, in order to extricate themselves from which it has been thought desirable to examine minutely certain methods of analysis, more or less novel, as applied to the detection of adulterants in butter.

The particular point to which I have lately devoted a considerable amount of attention is, as indicated by the title of my paper, the question of the iodine absorption of butter fat, and although the results which I have obtained have not proved absolutely constant and conclusive, still I believe they are of sufficient interest and importance to justify me in bringing them under the notice of the members of this Society.

Before giving a description of my own results I ought, perhaps, to make a brief reference to a paper on "The Analysis of Fats and Oils," read by Muter and De Koningh at the February meeting of the Society of Public Analysts, the first part of which is published in the current number of the Analyst. I was not present at the meeting in question, but I gather from the partial report which has already appeared that the iodine absorption of the fatty acids, separated from butter fat by a special process, was one of the points dealt with. As my determinations of the iodine absorption were, however, all made on the pure butter fat, and not on certain fatty acids produced therefrom, as in the case of Muter and De Koningh's experiments, our respective results are perhaps hardly comparable.

A short time ago, when I examined the most likely books of reference, in the hope of coming across work of a similar nature by other chemists, the only iodine absorptions of butter fat of which I could find any record are those given in Allen's "Commercial Organic Analysis" (vol. ii., p. 50), and I was surprised at the discrepancies between the results there stated.

According to Hübl, the iodine absorption of butter fat varies from 26·0 per cent. to 35·1 per cent., while Moore found it to be from 19·5 per cent. to 38 per cent. These figures are so widely divergent that, if such extreme variations were likely to occur in the case of genuine butter, it is evident the iodine absorption test would be of comparatively little value as a means of detecting adulteration.

However, I determined to investigate the matter more fully myself, by ascertaining the iodine absorption of a number of specimens of butter, the genuine character of most of which I can vouch for, and also that of samples which were received for analysis in the ordinary way of business. The estimations were all made under as nearly as possible the same conditions. About ten grains of the filtered fat of each sample was dissolved in 300 grains of chloroform; then a measured quantity of Hübl's reagent (usually 400 grains) was added, and the mixture allowed to stand four hours. At the end of that time 300 grains of a 10 per cent. solution of potassium iodide and 1,000 grains of water were added, and the excess of iodine determined by means of decinormal thiosulphate solution. A blank experiment was, of course, always made at the same time, with exactly the same quantities of the various reagents.

In the following table will be found some of the results which I have obtained by the examination of butter, margarine, and mixtures of the two in this manner :—

BUTTERS.

No.		Iodine Absorption. Per Cent.
1	English	33·62
2	„	36·31
3	„	35·09
4	„	35·30
5	„	33·83
6	„	32·35
7	Irish	39·42
8	„	37·17
9	„	38·91
10	Danish	34·11
11	„	34·69
12	„	38·70
13	„	40·36
14	„	35·74
15	„	34·64
16	„	34·01
17	„	33·68
18	„ (very fine quality)	38·91
19	German	35·49
20	„	34·51
21	„	36·21
22	„	38·25
23	„	35·01
24	„	23 60

BUTTERS—*continued.*

No.		Iodine Absorption. Per Cent.
25	Swedish	39·11
26	„ (said to be old)	35·10
27	Finnish	32·41
28	„	33·36
29	French (stale Brittany)	32·63
30	New Zealand	37·83

MARGARINES.

No.		
1	Ordinary	63·81
2	„	63·95
3	„	62·29
4	Good	63·85
5	„	65·82
6	Cheap	71·51
7	„	75·22

MIXTURES.

No.		
1		50·27
2		50·24
3	Better than above	49·26
4	High priced	56·65

The above list contains representative samples of butter from several countries, though it is by no means as complete as I should have wished. An examination of e figures given in the table will show that, with one exception, the iodine absorptions all the samples of butter fat were fairly concordant, the mean of thirty determina- ons being 35·34 per cent., with extreme limits of 23·6 per cent. and 40·3 per cent.

I am unable to give any satisfactory explanation of the former result. It was elded by a butter, the genuine character of which I have no reason to doubt, judging th by the source whence it came and also by the results which were obtained on amining the sample by the usual methods of butter analysis.

The sample with the highest iodine absorption (40·3 per cent.) which I have corded in the table was also, to the best of my knowledge, genuine butter, but, as only partial analysis was made, I am unable to speak with absolute certainty upon this int.

Another sample, said to be German butter several months old, which was sub- itted to me for examination, had an iodine absorption of 52·4 per cent. The other alytical results obtained during the examination of this sample led me to the conclusion at margarine was present, and I reported to that effect.

It will, of course, be readily understood that I had a special desire to apply the dine absorption test to butter known to be genuine beyond all possible doubt, but pos- ssing decidedly abnormal properties, in order to ascertain whether the iodine absorption as affected to any extent.

Several samples which I had at hand gave more or less abnormal results as regards e volatile and soluble fatty acids, etc., but, strictly speaking, I could not guarantee eir absolute genuineness.

A couple of months ago I accordingly wrote to Mr. Allen, asking him to kindly pply me with a small quantity of the abnormal butter referred to in his paper of ecember last. Mr. Allen informed me that he had none of the butter left, but as he ad sent some to Mr. Estcourt, he advised me to apply to that gentleman, which I did. r. Estcourt was, however, unable to let me have any of his sample, so I have unfortu- ately not been able to carry out my intention in connection with a genuine abnormal utter.

This fact notwithstanding, I believe the figures which I have tabulated in this aper will in many cases be of considerable value to my brother analysts, and it is ntirely with that object in view that I now publish the results of my experience with this particular process.

It has been suggested to me by a well-known butter merchant, for whom many of the analyses recorded above were made, that in summer the iodine absorption of butter may vary to a greater or lesser extent from that of butter produced in the same districts during the winter months.

This is a point which remains to be investigated, and if the results obtained are of sufficient interest, I shall be glad to bring them before the Society on some future occasion.

"PURE" CHEMICALS.

By Bertram Blount.

(*Read at the Meeting, April*, 1889.)

It is said that a bad workman finds fault with his tools, and the statement can hardly be disputed. It is, however, commonly found that a tacit deduction is made to the effect that he who finds fault with his tools is a bad workman, the fact that the converse of a true proposition is not necessarily true, being ignored. The resulting fallacy is frequently apparent, but in no instance is it more conspicuous than in the case of the chemist, where he is the bad workman, who is slow to find fault with and strive to improve his tools, wherever it is possible to do so.

Alive to the stress rightly laid by chemists on the importance of using reagents of the utmost attainable purity, manufacturers have long been in the habit of selling chemicals claiming to meet this requirement, and to distinguish them from less pretentious preparations have appended to them the titles "pure," "purif," "puriss," "chemically pure," "pure for analysis," and other reassuring terms. Rash is the analyst who relies on them, if anyone do. There is certainly usually a perceptible difference between them and the "commercial" kinds, but it is seldom absolute, and often wholly external. Much attention is paid to the appearance, packing, and above all to the labelling of such products, but with these their claims for consideration too often end, and one finds that if one desires something approaching purity, one must make it oneself.

A feeling not remote from disgust is produced, and leads the chemist to name his own preparations more modestly. Something of the sort probably induced Muir and Adie, in their recent paper on "The Interaction of Zinc and Sulphuric Acid" (*J. Chem. Soc.*, 1888, 47-58), to call their own admirable metal "approximately pure zinc," in contradistinction to the high-sounding titles used by the manufacturers from whom they obtained their raw material.

Similarly one finds in most laboratories the labels of reagents that have been prepared by the chemist himself, confining their assertions to some specific impurity, *e.g.*, "Hydrochloric Acid free from Arsenic," or particular process, such as "Calcium Carbonate for Lawrence Smith's alkali process."

If one be unwilling to undertake the tedious labour of preparing everything one has a difficulty in buying pure, and write to the maker supplying the offending article, mentioning its impurity, one usually finds that he is in no way disconcerted, but serenely admits that such is its normal condition. On further pressing he will perhaps confess that the hypercritical may detect room for improvement, and possibly volunteer the statement that he occasionally makes a small quantity of a still higher degree of purity, with an, if conceivable, more elaborate name—at a correspondingly higher price. On receipt of this it is commonly found that the improvement effected is by no means startlingly great.

As an illustration of the sort of thing I mean, I may quote a few figures from those obtained in the course of my own practice, in examining some of the reagents most largely used in the laboratory:—

In two samples of "pure" hydrochloric acid were found 0·328 and 0·118 grams per litre of solid matter, respectively; in three of "pure" nitric acid, 0·212, 0·66, and 1·084 grms. per litre; in one of "pure" sulphuric acid, 0·100 grms. per litre; in "pure" ammonium chloride, 0·23 per cent. of non-volatile matter containing manganese and lead. In "pure" ferric chloride I have found arsenic in notable amount, quite sufficient to prevent its use in Clarke's process for the determination of that body in copper (*J. S. C. I.*, 1887, 352). While speaking of ferric chloride, I may mention that the method one sometimes finds given (notably in "Crooke's Select Methods," p. 432), for freeing it from arsenic by repeated evaporation with hydrochloric acid, is utterly fallacious; it is based on the assumption that the arsenic is in the arsenious condition, an assumption which is not only unwarrantable, but inherently improbable. Purification can be effected by evaporating repeatedly with hydrochloric acid in presence of a suitable reducing agent, such as ferrous chloride, which may be conveniently prepared *in situ* by the addition of some iron wire.

Returning to my own unpleasant experiences, I have come across chromic anhydride which, when heated in blank with sulphuric acid, evolved sufficient gas of some sort to cause the potash bulbs to gain weight appreciably, and thus prove itself unsuitable for the combustion of carbon in iron and steel. In connection with this, I may recall the fact that Allen has mentioned the same difficulty (Abstr. Proc. C.S., 56, 76), and overcomes it by preparing his chromic anhydride by the action of sulphuric acid on fused chromate of lead. This I have not tried, having for some time past abandoned the use of chromic anhydride in favour of the plan of burning the carbon in a porcelain tube, in a stream of oxygen from a cylinder of the compressed gas.

Though not strictly covered by the heading of this paper, I may mention that I have had to do with an iridio-platinum dish, which rapidly lost weight when heated empty in a muffle, with a stoneware jar which appeared to be lined with plaster of Paris, so persistently did calcium sulphate occur in the distilled water that was attempted to be stored in it, and with Nessler solution so insensitive, that sewage itself might feel secure from detection by it.

With regard to the practical bearing of all this: it may be urged that to insist on the necessity of examining reagents before using them, is to advance a platitude, and truly it is; but I submit that though such examination cannot be dispensed with, it ought not so constantly to lead to the detection of impurities. If a manufacturer undertake the preparation of pure chemicals, it is not too much to demand that his productions shall in some degree fulfil his promise. Certainly if a reagent be complained of, he will generally replace it; but the substitute is by no means necessarily any better than its predecessor, and, apart from that, the delay and annoyance suffered by the analyst, are in no way made good to him. It certainly appears to me within the pale of discussion as to whether the maker could not be sued for breach of the contract implied by the use of the terms "pure," "puriss," etc., on the label, and be cast in damages for any inconvenience or loss occasioned by a reliance on his statement.

Passing to suggestions for an improvement of such a state of things, the only one that occurs to me is some arrangement by which an enterprising maker, who was a thorough chemist, or employed one, could be induced to manufacture chemicals of as high a degree of purity as the analyst would prepare in his own laboratory, and with similar specific limitations. By this I mean that if freedom from all impurities could not with any effort, commercially possible, be attained, yet a freedom from the special impurities that interfere with the main use of the reagent under consideration should be sought, and it should be labelled to that effect. That this is not without precedent is shown by the fact that assayers are supplied with lead and litharge, free from the specific impurity silver, which make no claim to be considered pure in other respects.

Further, if it be desired to obtain a pure product from a manufacturer less ideal than the one mentioned above, it is necessary to make plain to him that the handsome appearance of any reagent is valuable only if it be an index of purity, and that it is, by itself, an altogether secondary matter.

Laboratory, Broadway, Westminster, S.W., April 9th, 1889.

SOME EXPERIMENTS WITH THE SOAP SOLUTION.

By Elwyn Waller, Ph.D., New York.

(*Read at the Meeting April 10th,* 1889.)

Some little time since I had occasion to examine some samples of water, with a view to determining their comparative value for boiler and manufacturing purposes. With one of the samples especially, which was very hard, the soap test gave such unsatisfactory results that the method described by O. Hehner (Analyst VIII. 77) was tried upon it, and in order to make the results comparable among themselves, the same method was applied to the other samples.*

The results on the two hardest samples were as follows (sample A being the one especially referred to):—

Hardness (= $CaCO_3$) in Parts per 100,000.

	By Soap Test.			By Hehner's Method.		
Sample.	Permanent 20 c.c.	Total 20 c.c.	Total 10 c.c.	Temporary.	Permanent.	Total.
A	41·915	45·955	51·050	21·14	39·65	60·79
C	31·31	33·30	34·845	19·20	19·93	39·13

Of course in the above, the 20 c.c. and 10 c.c. lots were diluted up to 100 c.c. with distilled water before applying the soap test.

Some of the other analytical results on these waters were as follows:—

Parts per 100,000.

	Analyst.	Cl.	SO_3	CaO.	MgO.	Total Solids.	Loss on Ign.
Water A.	Wallor ..	1·633	28·10	26·85	4·829	86·5	8·0
,,	Chemist C.	3·00	28·76	26·74	..	88·1	6·0
,,	,, H.	..	..	..	..	81·3	17·6
Water C.	Waller ..	1·100	12·02	15·31	3·280	49·0	8·0
,,	Chemist C.	..	..	..	..	60·7	5·0

---

* *Lakmoid* was found to be more satisfactory as indicator in this case, than the phenacetolin indicator, recommended in Mr. Hehner's article.

I did not learn until some time afterwards that other chemists had also made examinations of samples of these waters a month or six weeks before mine was made.

The results which they obtained for hardness by the soap test were as follows:—

PARTS PER 100,000.

		Temporary.	Permanent.	Total.
Water A ..	Chemist C.	18·8	29·7	48·5
"	Chemist H.	..	..	32·
Water C ..	Chemist C.	14·7	19·	33·7

As I had reported the results obtained by the Hehner method the discrepancy was very marked, especially in the case of water A.

Having noted that with different amounts of the water different results were obtained by the soap test, the idea naturally suggested itself to try the action of soap with different amounts of lime and magnesia. Accordingly a solution of pure double refracting spar was made after the regular method (solution of 1 grm. in hydrochloric acid, evaporating until neutral, and dilution to 1 litre). (This will be called "Ca solution.") A solution containing 2·46 grms. crystallised magnesium sulphate in 1 litre was also made (called "Mg solution"). A third solution, made by mixing 150 c.c. of each of the other solutions, was also prepared (called "mixed solution"). Different proportions of these solutions, diluted in every case to 100 c.c. with distilled water, were then tested with the soap solution.

The results were as follows:—

c.c. of Solution diluted to 100 c.c.	Cubic centimeters of Soap Solution to Produce Permanent Lather after Standing.							
	Ca Solution.		Mg Solution.			Mixed Solution.		
5	10·7	10·8	12·15	12·35	12·20	10·2	10·2	10·2
10	19·7	19·9	23·5	23·3	..	18·3	..	19·7
20	37·7	37·8	(35 to 40 uncertain.)			(27·5 uncertain.)		
30	54·	55·	..	..	..	..	..	..
40	(74 uncertain.)		..	..	..	..	..	..

100 c.c. of distilled water required 1·3 and 1·4 c.c. soap solution to form a lather.

The figures in the last column for "mixed solution," were obtained by first adding 70 or 80 per cent. of whole amount of soap solution, which was finally used, mixing in without violent shaking, allowing to stand for 30 minutes, and then finishing the titration as usual. Where magnesium was present, care was taken to carry the test beyond the point where a temporary lather forms, mentioned by Wanklyn, and probably noticed by every chemist who has made much use of the soap test.

My usual practice has been to take the standard of the soap solution from the results obtained on 10 c.c. of the Ca solution, making no allowance for the action on the soap of the water used for dilution. That mode of calculating has seemed to give the most accurate results that the soap test appeared capable of. Calculating the standard in this way, the value of 1 c.c. of the soap solution would be 0·01 ÷ 19·8 = 0·000505 $CaCO_3$, but it is evident that a variety of different standards could be obtained from the above results on the Ca solution. Two different sets of standards would be obtained according as we deduct 1·35 c.c. for the influence of the water of dilution or not.

In the following tables, the columns headed "Ded't" give the results obtained by calculation, where this deduction is made :—

Ca Solution.

	Soap required by 10 c.c.		Standard 1 c.c. Soap = $CaCO_3$.	
Test made on		Ded't.		Ded't.
5 c.c.	21·5	18·8	0·000465	0·000532
10 c.c.	19·8	18·45	0·000505	0·000542
20 c.c.	18·825	18·2	0·000530	0·000549
30 c.c.	18·17	17·71	0·000550	0·000564

The hardness of the Mg solution and mixed solution, expressed in terms of $CaCO_3$, would be as follows :—

	Mg Solution.				Mixed Solution.			
	10 c.c. = Soap.		Hardness = $CaCO_3$ per c.c.		10 c.c. = Soap.		Hardness = $CaCO_3$ per c.c.	
Test made on.	c.c.	c.c. Ded't.	Standard 0·000505.	Standard 0·000542. Ded't.	c.c.	c.c. Ded't.	Standard 0·000505.	Standard 0·000542. Ded't.
5 c.c.	24·46	21·76	0·0012352	0·0011794	20·4	19·7	0·00103	0 0010677
10 c.c.	23·4	22·05	0·0011817	0·0011951	18·3	16·95	0·000924	0·009187
10 c.c. st'dg.	..	..	..	..	19·7	18·35	0·000995	0·009946

These results confirm the observations of Mr. A. H. Allen (December Meeting of the Society of Chemical Industry, vol. VII., p. 795), that the ratio of lime hardness to magnesia hardness by the soap test is not 2 to 3, or 42 to 75, as asserted by Wanklyn (*Water Analysis*, 7th Ed., p. 100); also that a mixture of calcium and magnesium salts shows a lower degree of hardness than either of them separate.

What remained of the sample of water A was then taken up after it had been standing 3½ months. It was again tested for total solids, hardness by Hehner's method, etc. The results were :—

	Total Solids.	Loss on ign.	Temp. Hard's.	Perm. Hard's.	Total Hard's.
When first received	86·5	8·	21·14	39·65	60·79
After standing 3½ months ..	77·	9·	8·2	36·2	44·4

Some of this water was placed in a bottle, and tests were applied, as with the Ca and Mg solutions, diluting whatever amount was taken, up to 100 c.c., with distilled water before applying the soap test.

Experiments on Sample A (after standing 3½ months).

cc. of a taken.	c.c. of Soap required.		100 c.c. of a=c.c. Soap		Hardness per 100000.	
		Ded't.		Ded't.	Standard =0·000505.	Ded't. Standard =0·000542.
1	1·6	0.25	16·	2·5	80·800	13.550
2	2·6	1·25	13·	2·25	65·650	33·875
5	5·1	3.85	10·2	7·7	51·510	41·734
10	8·75	7·45	8·75	7·45	44·187	40·379
20	14·4	13·05	7·2	6·525	36·360	35·365
30	19·8	18·45	6·6	6·15	33·30	33·333
40	26·2	24·85	6·55	6·2125	33·077	33·672
50	31·4	30·05	6·28	6·01	31·714	32·574
60	37·4	36·05	6·23	6·008	31·478	32·573

Which figure should be taken as representing the total hardness of this water?

The soap solution was entirely used up in making these tests, or they would have been carried farther. A small amount of another soap solution happened to be available, which like the first had deposited a slight sediment, which as before was filtered off, affording a clear, bright solution. With this tests were also made on the Ca solution. The results were:—

Ca Solution used.	Soap Solution Used.			10 c.c.=c.c. Soap.	
					Ded't.
5	10·85	10·85	10·9	21·73	21·23
10	20·5	20·6	21·	20·3	19·45
20	37·	38·1	38·	18·85	18·21
30	56·	..	..	18·67	18·25

100 c.c. of distilled water took 1·2 and 1·3 c.c. soap solution.

The value of the soap solution was a little different from the first one, but the results show essentially the same as before.

The results by the soap test are apparently altogether unreliable, for very hard waters especially, and even with waters comparatively soft the results for permanent hardness are still more untrustworthy. This last point is alluded to by Mr. Wanklyn (*Water Analysis*, 7th Ed., p. 98) where be expresses disapproval of any attempt to determine the different kinds of hardness (temporary or permanent) by means of the soap test.

It would seem advisable to abandon the use of the soap test entirely, for although all *chemists* are well aware of the roughly approximate character of the results, their clients usually are not, and attribute as much importance to the figure for "hardness by soap test" as to any other figure on the report of a chemist whom they may employ.

APPARATUS FOR BUTTER ANALYSIS.

By H. Droop Richmond.

In a paper, a translation of which appeared in the Analyst for November and December, 1888, and January and February, 1889, Wollny recommends the use of a 50 per cent. soda solution for saponifying the butter fat in Reichert's process, and the following apparatus has been found useful for storing this solution.

It consists of a bottle holding the solution fitted with an indiarubber cork, through which two tubes pass, one of them, open at both ends, carrying the blowing-ball A, and the other being bent over and terminating in the pipette B. The tube passes down inside the pipette, and terminates at a mark, to which, if the pipette is filled, just over 1 c.c. of soda solution would be discharged. In the upper part of the pipette at tube D, on which is the tap C, is joined at right angles, to the end of which a soda-lime tube is attached (not shown in figure). At the bottom of the pipette a cap (either of glass as in figure, or preferably of indiarubber) is fitted.

The apparatus is used in the following manner: On compressing the ball A, the soda solution is forced up the other tube into the pipette, the air escaping by the tube D (the tap being open). When the pipette is full to the mark, or a little over, the pressure on the ball is released, and the excess of soda solution, together with some air, is sucked back. The tap is now turned off and the cap removed, and the flask or other receptacle placed under the bottom of the pipette; on the tap being turned, the caustic soda solution runs out, and the cap being replaced, the apparatus is ready for use again.

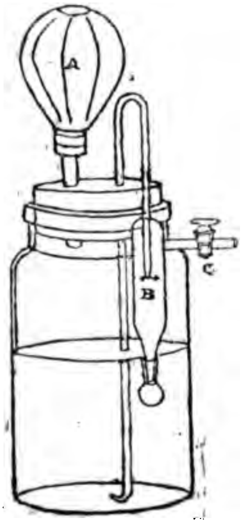

Fig. 1.

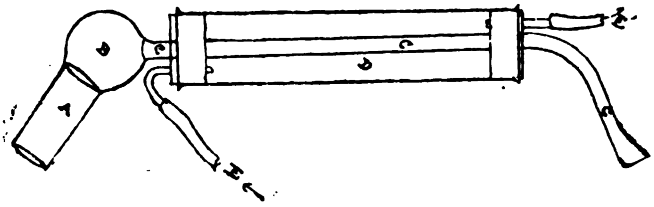

Fig. 2.

As long as the level of the caustic soda in the pipette is not allowed to rise as high as the tube D, no particular care need be taken to fill to any place, the excess being invariably sucked back. All the air that enters the apparatus must pass through the soda-lime tube attached to D, and is thus free from carbonic acid. The quantity 1 c.c. has been chosen, as it is sufficient for 2½ grs. of butter-fat, the quantity taken for the Reichert process; should 5 grs. be used, 2 c.c. may, of course, be taken.

Another piece of apparatus which will be found useful is this (Fig. 2), which is used when distilling. It consists of a tube, A 1 c.m. in diameter, and about 4 c.m. long, connected by a bulb, B 2 c.m. in diameter, with a tube C about ·6 c.m. in diameter and 15 c.m. long, at angle of about 120°; this at the end is also bent at 120°. On this tube C is fitted a small condenser D, the water running in at E and out at E′. The distilling flask is attached by a cork to A, and the end of C is inserted into a condenser, the water being allowed to run freely through D until the fatty acids are melted. When this is the case, *the water is removed from D* and the distillation proceeded with. The bulb B prevents any spurting that may take place, and the wide tube A allows the condensed liquid to run back into the flask without trouble. If care be taken to remove the water from D, the results are not sensibly different from those obtained with the Wollny apparatus.

(*Conclusion of the Society's Proceedings.*)

ESTIMATION OF SOLUBLE AND INSOLUBLE FATTY ACIDS IN BUTTER

By William Johnstone, Ph.D., F.I.C., F.C.S.

Of the butter to be examined, 2·5 grammes of the clarified fat is saponified with a known quantity of alcoholic potash in a closed flask with the aid of heat and repeated shakings.

When saponification is complete, the soap solution is transferred to a suitable vessel and titrated with standard acid, and thereby the amount of KHO ascertained which is required to saponify the 2·5 grammes of the following three samples of butter.

Thus—	A.	W.	Y.
Alkali taken $\frac{N}{KHO}$	25·00 c.c.	25·00 c.c.	25·00 c.c.
Acid required	14·78 c.c.	14·96 c.c.	14·76 c.c.
$\frac{N}{KHO}$ Consumed	10·22 c.c.	10·04 c.c.	10·24 c.c.

The alcohol is now boiled off, excess of acid added so as to decompose the soap, and then gently heated until the fatty acids are melted.

The insoluble fatty acids are now filtered off through an unweighed filter in the usual manner, and then thoroughly washed with boiling water until the washings are neutral, when the filter containing them is set aside in a moderately warm place and allowed to air dry. When the filter is sufficiently dry, it is taken and folded up and transferred to a Soxhlet extraction tube and thoroughly extracted with *dry ether*, the acids being received in an accurately weighed flask.

When the extraction is complete, the ether is evaporated off, and the flask and contents put in the water oven, and when dry allowed to cool and finally weighed.

Thus—

	A.	W.	Y.
Fatty acid + Flask	29·1917	29·1346	29·8400
Flask	26·9430	26·8844	27·5942
Fatty acid (insoluble) in 2·5 grammes	2·2487	2·2502	2·2458
× 40 = Per Cent.	89·9480	90·0082	89·8320

When the percentage of insoluble fatty acids have been ascertained, add sufficient standard alkali to the flask, heat gently when saponification rapidly takes place, then wash the soap solution thoroughly into a basin and titrate again with standard acid and estimate the amount of alkali now consumed by the insoluble fatty acids.

Thus—

	A.	W.	Y.
$\frac{N}{KHO}$ Taken	20·00 c.c.	20·00 c.c.	20·00 c.c.
Acid required	11·87	11·74	11·94
$\frac{N}{KHO}$ Consumed	8·13	8·26	8·06
Then—			
$\frac{N}{KHO}$ Consumed by 2·5 grammes butter	10·22 c.c	10·04 c.c.	10·24 c.c.
$\frac{N}{KHO}$ Consumed by insoluble fatty acids	8·13	8·26	8·06
$\frac{N}{KHO}$ required by soluble fatty acid of 2·5 grammes..	2·09	1·78	2·18

The percentage of soluble fatty acid (as butyric) by Reichert-Wollny's process is:—

4·37	4·44	4·68

The amount of soluble acids found in the above three samples of butter calculated as butyric acid:—

	7·35	6·26	7·67
Deduct that found by distillation	4·37	4·44	4·68
	2·98	1·82	2·99

Working in the manner above described, we get an accurate estimation of the amount of the total soluble fatty acids contained in a sample of fat, and it is, therefore, an infinitely superior process to Reichert's, for by the latter only a proportion of the soluble acids are obtained, and that merely of the *volatile acids*; besides, the Reichert-Wollny method only furnishes comparative results when rigidly performed in the same manner, a process which ought not to be tolerated when there is another method which gives with absolute accuracy the total amount of soluble fatty acids. Reichert's process certainly allows the operator to make a very rough estimation of the volatile acids, but it does not give him any insight as to the composition of the volatile acids, viz., as to the proportion of Butyric, Propionic, Acetic, and *Formic* acids, all of which are sometimes present in the distillate, and last, but not least, the proportion of non-volatile soluble fatty acids.

The following equations are extremely interesting, and may very easily be brought about with ordinary glycerine.

$$C_3H_8O_3 + 2KHO = KC_2H_3O_2 + KCHO_2 + H_2O + 2H_2$$

$$C_3H_8O_3 + KHO = C_3H_5KO_2 + 2H_2O$$

What must it be with nascent glycerine in presence of excess of KHO ?

RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Test to Distinguish Resorcin from Carbolic or Salicylic Acid. H. Bodde *Nederl. Tydschr. v. Pharmacie*, etc., May, 1889.—If a few drops of a solution of sodic hypochlorite are added to a watery or alcoholic solution of resorcin, a violet colour, rapidly changing to yellow, is produced. On warming or adding excess of the reagent, the liquid gets dark brown. One part of resorcin dissolved in 10,000 parts of water will still show the reaction. Carbolic, salicylic, benzoic and other allied acids do not show the reaction, and at the most turn only slightly yellow on warming. Pyrocatechin, when treated with the reagent, turns a vanishing green; hydroquinon soon gets yellow and red. Another test is to first add a little liquor ammonia and then a few drops of the hypochlorite, when the liquid will give a reddish violet colour, which changes green on boiling. The colouring matter is not taken up by benzol. The reaction is not shared by salicylic acid, benzoic acid or antifebrin, but carbolic acid thus treated turns a greenish blue, which is partly decomposed by benzol. The colours are changed to red by dilute sulphuric acid.

L. de K.

Composition of Macassa Oil. L. van Itallie.—*Nederl. Tydschr. v. Pharmacie*, etc., May, 1889.—The seeds of Cussambrium spinosum contain about 36 per cent. of a soft fat easily soluble in ether, petroleum spirit, and benzol, but slightly so in alcohol. On warming, the bulk of the fat melts at 22° C. The author obtained on analysis the following results: Iodine number 53. Insoluble fatty acids, 91·4 per cent. Specific gravity of these acids, ·922 at 15° C.; their melting point, 55° C. One gram. of the fat contained free fatty acid corresponding with ·0166 gram. caustic potash. The saponification equivalent was found to be 230. The glycerin was estimated by Hehner's bichromate method, and amounted to 6·3 per cent. The volatile acids were found to be acetic and butyric acids. The presence of oleic acid was proved by dissolving out the lead soap with ether and weighing the lead oleate. Some of the oil was saponified and the acids thrown up with hydrochloric acid. They were then treated with alcohol of 70 per cent. to remove bulk of the oleic acid, and then treated with alcohol of 90 per cent. The insoluble portion proved to be arachidic acid, whilst the neutralised solution gave with magnesium acetate a precipitate which, from its percentage of magnesia and other properties, was found to be a salt of lauric acid. The fat therefore consists of the glycerides of lauric, oleic, arachidic, acetic, and butyric acids. A sample of the oil which had been kept for twenty years had not in the least deteriorated.

L. de K.

Estimation of Alkaloids in Coca Leaves. Prof. Van der Marck. *Nederl. Tydschr. v. Pharmacie*, etc., April, 1889.—The author first tried Squibb's method. Fifty grammes of the leaves were macerated with 40 grammes of 95 per cent. alcohol and 1·6 gram. of hydrochloric acid. After percolating and distilling off the spirit, the residue was taken up with 30 c.c. of ether, which was then shaken with 30 c.c. of water and 1 c.c. of 10 per cent. acid. The watery fluid was drawn off, and the ether once more shaken with 10 c.c. acidified water. The mixed acid fluid, after being once more shaken with a little ether to remove any colouring matter, was rendered alkaline with sodium carbonate, and the isolated alkaloid shaken out with ether. The author obtained ·49 per cent. of alkaloid, but objects to the process on the following grounds: 1. The quantity of alcohol ordered is too small, and should be doubled. 2. As a rule, the ether, when shaken with acid water, forms a troublesome emulsion. 3. Twice shaking with ether is insufficient. 4. The residue left on evaporation is not pure cocain, as it did not dissolve completely in hydrochloric acid, and left about 50 per cent. of insoluble residue. It is also dangerous to distil a strongly acid alcoholic solution of cocain, as it so readily decomposes. The author next tried the process of Castaing. Fifty grammes of coca leaves were treated with 400 c.c. of boiling water, and after standing for half an hour percolated. The residue was then percolated with alcohol of 85 per cent., and the mixed filtrates thrown down with lead acetate. After the excess of lead had been removed with sodium sulphate, the liquid was concentrated, filtered, rendered alkaline with sodium carbonate, and shaken out with ether. The amount of cocain was ·38 per cent., but it was only partly soluble in dilute hydrochloric acid, and much coloured. The next process tried was that of Albertoni and Guareschi. Fifty grammes of the leaves were exhausted with ether, the ether was distilled off, the residue treated with boiling water, and the solution evaporated to dryness with a little magnesia. On extraction with amylic alcohol, ·14 per cent. of alkaloid was obtained. As a last resource, Bignon's process was tried. Fifty grammes of the powdered leaves were macerated for forty-eight hours with a 20 per cent. solution of sodium carbonate, then dried on the water-bath and exhausted with petroleum spirit to dissolve the cocain. The solution was shaken with dilute hydrochloric acid, which takes up the cocain. The acid fluid was then rendered alkaline and shaken with ether, which extracted the cocain. The percentage of cocain got by this process amounted to ·06, a proof that most of the alkaloid had been decomposed by the strong alkali. The author then tried the following idea: 25 grammes of the powder were mixed with 10 grammes of magnesia and a little water, and dried on the water-bath. After percolating with ether (so as not to dissolve any hygrin), the ether was distilled off and the residue treated with acid water and filtered. After rendering alkaline, the alkaloid was extracted with chloroform. Obtained ·2 per cent. alkaloid. A similar experiment was made with 50 grammes of the leaves, and ·19 per cent. of alkaloid was obtained. Ether may, however, be advantageously substituted for chloroform. The author finally recommends the following process: 50 grammes of the powdered leaves are mixed with 20 grammes of magnesia, and dried at 60° C., then extracted with ether. After distilling off the ether the residue is taken up with 30 c.c. of 2 per cent. hydrochloric acid. The acid is filtered and repeatedly agitated with ether, to remove colouring matters. Ammonia is then added, and the mixture shaken out three times with 25 c.c. of ether. After standing for a little while over a few pieces of calcium chloride, the ether is evaporated and the alkaloid weighed.

L. DE K.

ESTIMATION OF DEXTRIN IN NARCOTIC EXTRACTS. PROF. VAN DER MARCK. *Nederl. Tydschr v. Pharmacie*, etc., April, 1889.—The author tried Pannetier's process, which arently leaves nothing to be desired, and is performed as follows: Two grammes of extract are dissolved in 50 c.c. of water and precipitated with 5 c.c. of liquor mbi. The filtrate is freed from lead by sulphureted hydrogen, and concentrated to -fifth of its bulk. An equal volume of spirits of wine is next added, which precipi- s dextrin, and some alkaline salts. The precipitate is washed with alcohol, dried weighed. To make sure, the dextrin may be inverted and estimated by Fehling's tion. Feeling no confidence in the method, the author tried the following experi- ts: Two grammes of extract of belladonna were dissolved in 50 c.c. of water, ipitated with liquor plumbi and filtered. After freeing the filtrate of lead and entrating, alcohol was added and the fluid remained clear. Two mixtures were made up containing respectively 5 and 10 per cent. of dextrin, but alcohol failed roduce a turbidity. With a mixture containing 15 per cent. of dextrin, a faint d was obtained, and, when 20 per cent. had been added, about one-third of the trin was recovered. On repeating the last experiment, but with addition of much e alcohol, 67 per cent. of the dextrin was recovered. The author, therefore, con- led that the method is perfectly useless, and endeavoured to find a better way. It f course, perfectly well known that dextrin, when heated with a 2 per cent. strong rochloric acid is gradually converted into glucose. Before applying the inversion ess, it was necessary to prove: 1. Whether the extracts themselves contain copper cing bodies; 2. Whether the inversion can be quantitatively depended on. Two mmes of belladonna extract were dissolved in 10 c.c. of water, and made up with hol to 75 c.c. The precipitate was filtered off and washed with alcohol of 75 per . The alcohol was evaporated, and the residue treated with Fehling, which was ly reduced. Fehling's solution was also reduced by the watery solution of the ipitate. Two grammes of the same extract were precipitated with liquor plumbi, filtrate freed from lead by passing hydrogen sulphide, and after boiling neutralised. liquid strongly reduced the Fehling. As these experiments proved that the extract ained reducing bodies, it became important to know the reducing power of different acts from various origins. The author, therefore, took samples of extract of bella- na (Nos. 1, 3, 5, 7) and extract of hyoscyamus (Nos. 2, 4, 6, 8). Two grammes were lved in 50 c.c. of water and precipitated with 8 c.c. of liquor plumbi. After oving the lead, the liquid was evaporated down to 20 c.c. and titrated with Fehling's er solution. It was now found that 10 c.c. of the filtrate (=1 grm. extract) ired of Fehling:—

Nos. 1.	2.	3.	4.	5.	6.	7.	8.
·7	·5	1·3	2·2	1·4	1·8	1·2	1·9 c.c.

esponding with an average percentage of ·8 per cent of glucose, which may be dis- rded. The next step was to ascertain whether dextrin may be completely converted glucose. Ten grammes of the best commercial dextrin were dissolved in 100 c.c. of ter. Ten c.c. of this solution took an average of 9·6 c.c. of Fehling, corresponding with er cent. of glucose. The sample lost in drying 11·6 per cent. of water; 2 grms. of sample was dissolved in 100 c.c. of 2 per cent. hydrochloric acid, and heated for two urs in water of 90° C. After cooling, the liquid was made up to the mark and then ated. 10 c.c. of Fehling required 23 c.c. of the liquid, which proved a loss of 8·8 per cent. dextrin. In a second experiment, when the heating was continued for three hours, the loss amounted to 18·9 per cent.* These results are, however, more satisfactory than those obtained by Wertha and Payer, who respectively got a loss of 58 and 38 per cent. The author thinks, however, that this process may be still found useful in cases of serious frauds.

L. de K.

Copper in Gherkins. Wesener. *Zeitschr. f. Angew. Chemie*, No. 5.—One sample possessing an unnatural green colour gave on analysis ·0036 per cent. of copper oxide, or ·058 per cent. on the dried substance. A second sample of a yellowish-green colour only showed traces of copper, whilst a third sample seemed to be quite free from that metal. The copper in the first sample was no doubt the cause of the serious illness of a child. It will be interesting to analysts to read that not a trace of copper could be detected in the preserving vinegar.

L. de K.

Estimation of Alkaloids in Extracts. L. van Itallie. *Nederl. Tydschr. v. Pharmacie*, etc., January, 1889.—Five grms. of the extract are dissolved in water with the aid of 10 drops of dilute sulphuric acid (1—20), and then made up to 50 c.c. After standing for an hour or two with occasional shaking, 25 c.c. of solution of lead acetate (1—10) are added. After settling, the fluid is filtered through a dry filter. 50 c.c. of the filtrate are now mixed with 10 c.c. of the dilute sulphuric acid, and again filtered. From this second filtrate 50 c.c. are put into a separating funnel, and after rendering alkaline with ammonia, shaken out three times with 50 c.c. of chloroform. The chloroform is distilled off, and the residue, after dissolving in 5 c.c. of spirits of wine, titrated with $\frac{n}{100}$ hydrochloric acid, 1 c.c. of which represents ·00289 grm. atropine or hyoscyamine, ·0053 aconitine, or ·00127 coneine, lackmoid being used as indicator.

L. de K.

Analysis of Sulphate of Soda. Isbert and Venator. *Zeitschr f. angew Chemie, No.* 3, 89.—The anlaysis of commercial sulphate of soda is very troublesome, and consequently highly charged for by analysts, which causes the manufacturers to send but very few samples. The authors have found a very accurate and simple process, which suits the manufacturers. It is as follows: About 2 grammes of the samples are dissolved in the smallest quantity of hot water and mixed with a little ammonia and ammonium carbonate as long as a precipitate forms. The precipitate must be freed from soda by re-dissolving in hydrochloric acid and reprecipitating with ammonium carbonate. The mixed filtrate and washings now contain sodium sulphate, ditto chloride, and also the excess of sulphuric acid the sample might have contained. The liquid, which will measure about 100c.c., is evaporated in a platinum dish, to dryness. The residue is gently ignited to expel ammoniacal salts, and then weighed as pure sodium sulphate, any sodium chloride having been decomposed by the ammonium sulphate. If the sample should be rich in chlorine, a little extra ammonium sulphate should be first added to the filtrate. From the weight of the sodium sulphate is deducted the weight of sodium sulphate corresponding with the sodium chloride the sample contained, and which is estimated with deci-normal silver. The test analyses are very satisfactory.

L. de K.

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* See also Analyst, 1887, p. 138. L. de K.

LAW NOTES.

Borough Analyst v. The Bedford Corporation.

as an action in which Dr. Voelcker, of London, the Borough Analyst, claimed £21 from the
rporation for fees for making analyses. Mr. F. Low (Solicitor to the Society of Public
appeared for the plaintiff, and Mr. Bonsey (instructed by the Town Clerk) for the
.

w, in opening the case, said no doubt his Honour was surprised that a public official of the
Bedford should have to come to that Court to recover work done as public analyst, but
ysts generally, and particularly the Bedford analyst, were not popular officials, and Dr.
the former borough analyst, terminated his engagement under circumstances which probably
was aware of. [The Judge said this matter was perfectly immaterial to the action]. This
rial observation so far as his client was concerned, because he would not have taken the
t had it not been for the persuasion of the late Ald. Howard, who was a friend of his. Dr.
as appointed as public analyst to the Bedford Corporation in May, 1887, on the condition
to receive one guinea for every sample submitted for analysis. In the following November
cles were sent to him from Bedford for analysis, and among them were two samples of
the fourteen samples, thirteen were reported genuine, and no question could be raised on
o prosecution could follow. One sample of coffee was found to contain 80 per cent. of
d in reporting this case Dr. Voelcker did not use the form of certificate as prescribed by
d failed to insert the weight of the sample quite by accident, and in consequence of that
ation said it was useless to them as they could not take proceedings on it against the owner
ple. But while refusing to pay for this one sample, the Corporation also refused to pay for
fourteen samples. With respect to the blank left for the weight, a foot-note on the
said that when the sample could not be conveniently weighed the weight need not be
nd he contended that it was perfectly immaterial, but it would not be so with some
. The omission of the weight in this instance did not render the certificate useless, and he
that it was a good and sufficient statutory certificate under the Act. In the case of a
, in case the Court did not accept the certificate, the presence of the Analyst would be all
ecessary, and he informed the Corporation he would be ready to attend if wanted. He also
mend the certificate, but the Corporation said that after the certificate had once left his
ould not alter it. Six guineas had been offered by the defendants, but plaintiff would not
ecause he was entitled to twenty guineas, six other samples having later on been analysed.
dge said the point was as to whether the insertion of the weight was material in this

elcker stated that he weighed the sample of coffee, and it weighed 5oz. In all cases of
coffee, the weight was perfectly immaterial to the analysis, however small the sample
In some substances, such as drugs, weight was material.
examined: Had the sample been smaller, he should have retained it and asked for another
t for chemical reasons, but because it was always advisable to have a fair representative
t a very small sample from a large bulk might give a fallacious result. He examined every
truly stated the result in the certificates.
dge expressed his opinion that it was the intention for the weight to be inserted in all
pt where it could not be done.
nsey pointed out that one of the certificates was not signed, and was therefore useless. It
ed that in cases of articles liable to decomposition or change in constitution, mention must
it in the certificate, but in some of the certificates that came under this head this had not

w said it was unnecessary because all those samples were genuine.
nsey said that the object of the Act was that the form of certificate should be strictly
ith, and no proceedings could be taken before the production of the certificate properly
d signed.
dge was satisfied that the certificate must be made out according to the Statute, and after
left the analyst's hands he could not alter it.
nsey asked how they were to know that a proper sample had been sent if the weight was
The plaintiff had left out the material part by not inserting the weight, and the analysis
mplete until a proper certificate had been given.
onour thought some of the certificates informal, but reserved judgment, and Mr. Low
Honour to carefully consider the question of power to amend the certificate.

D QUESTION REVIVED.—A case of importance was heard at the Worship Street Police Court
ult., before Mr. Bushby, the presiding magistrate, against a tradesman who had been sum-
he instance of the Vestry of Bethnal Green for selling an article, to wit, coffee, adulterated
r cent. of chicory, contrary to the statute, and was, therefore, not of the nature, substance,
y demanded by the purchaser. Mr. Ogle appeared for the defence, and contended that his
protected by the notice printed on the wrapper, that the article sold was a mixture. Mr
of the Sanitary Department, who appeared in support of the summons, addressing the
, said this was the first case of the kind that had come under the notice of the Vestry, who

were desirous that it should be considered as a test case, and to have the decision of the Court upon it. The points to be considered were whether the sample was sold to the prejudice of the purchaser, as set forth in section 6 of the Act, and how far the vendor was protected by giving a label stating that the article was a mixture. It had been generally thought that if a customer asked for coffee the vendor might supply a sample containing any quantity of chicory, providing it is placed in a wrapper labelled, "A mixture of chicory and coffee." The wrapper produced by Mr. Ogle was itself an ingenious device; it was somewhat misleading, and was printed as follows: "The finest fresh-roasted coffee. This is sold as a mixture of chicory and coffee." It had been held by a metropolitan magistrate, in a recent case, that if coffee was asked for, the purchaser was defrauded if any other article was supplied, however it might be labelled. He would further call the attention of the magistrate to the case of Liddle *v.* Reese, and the judgment of Justices Lush and Manisty, the protection afforded to the vendor by the giving of a label, referred to in section 8 of the Act, applied to the sale of compounded articles of food mixed with any matter or ingredient not injurious to health, and not intended fraudulently to increase its bulk, weight, or measure. Coffee was not necessarily a compounded article, and where chicory was added, which could be purchased separately at about one-fourth the price, he respectfully submitted that the effect was fraudulently to increase its bulk and weight, and that, consequently, an offence had been committed against the statute. Mr. Bushby said, after hearing the evidence, that he was of opinion that the vender had complied with the statute by giving a label, and that the case could not be sustained. If, however, a point of law were submitted to him, he would consider whether he should state a case for an appeal to a superior court.

INTERESTING CASES OF THE ADULTERATION OF OLIVE OIL.—At the Wandsworth Police Court, on the 17th ult., J. Palmer and Sons, Balham, were charged by Inspector Smith with selling olive oil so adulterated as to be indigestible, and consequently dangerous to health. The Inspector produced Dr. Muter's certificate, which showed that the oil was chiefly mineral oil, so prepared as to resemble olive oil in appearance. The defendant was fined £10 and costs.

At the same court, on the 1st of May, W. Taylor, jun., of Clapham, was charged by Inspector Smith with selling adulterated olive oil. Dr. Muter's certificate showed the article to contain not less than 50 per cent. of ground nut oil. The defendant pleaded entire ignorance, and the magistrate seeing that the article was not dangerous to health, let him off with 1s. fine and costs.

At the Southwark Police Court, on Wednesday, the 15th ult., before W. Slade, Esq., E. R. Goodrich, wholesale and retail oilman, 35, Old Kent Road, appeared in answer to a summons taken out by John Edwards, the Inspector for the District of St. George the Martyr, S.E. (on the certificate of Dr. Muter), for selling olive oil adulterated with 20 per cent. of cotton-seed oil. The defendant's counsel stated that he would plead guilty as he had had the oil analysed and found the certificate to be correct. The defendant had called in all the oil he sent out as olive oil as soon as he knew it to be impure, and he urged the magistrate to impose only a small fine. The Inspector stated to his worship that it was labelled "Finest Lucca Oil." The magistrate imposed a penalty of £5, and 12s. 6d. as costs.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

"NOTES ON LACTOSE."

SIR,—I very much regret that I was prevented from attending the March meeting of the Society when Mr. E. W T. Jones read a paper upon "Lactose," as I certainly should have pointed out that a considerable portion of the results contained in his paper had already been published in the ANALYST, Vol. xi., page 27.

Mr. Jones' results, however, corroborate those obtained by myself. Thanking you beforehand for insertion of the above in your next issue. Yours, etc.,

WILLIAM JOHNSTONE.

13, Fish Street Hill, Eastcheap,
London, 6th May, 1889.

ABNORMAL BUTTERS.

To the Editor of the ANALYST.

SIR,—I was under the impression that Mr. Estcourt was himself the authority for my statement that he had habitually employed, with his Westphal balance, a thermometer-plummet of which the bulb was broken, and which, therefore, gave him only arbitrary figures. I presume I may take Mr. Estcourt's last letter as an indication that I misunderstood him, but the use of a broken thermometer-plummet for observations at the boiling point of water is quite compatible with the simultaneous possession of an undamaged plummet for use at ordinary temperatures.

I did not give up quoting specific gravities of melted fats at 100° F. at the suggestion of Mr. Estcourt; but I did adopt the use of the Westphal balance, in place of the Sprengel tube I had previously employed, at his suggestion. Mr. Estcourt employed a bath of heated paraffin, instead of one of boiling water as adopted by me.

ALFRED H. ALLEN.

May 14th, 1889.

THE ANALYST.

JULY, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

AN ordinary meeting was held at Burlington House on Wednesday, the 12th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

On the ballot papers being opened, it was announced that Mr. A. J. Hailes, F.C.S., had been elected as a member.

Mr. Cecil Howard, Public Analyst for the Strand District, was proposed for election as a member.

The PRESIDENT announced that the result of the ballot as to whether the summer meeting should be held in Paris or in Liverpool, was that the latter was preferred, and it would accordingly be held there, or in that district.

The following papers were read and discussed :—

"On Condensed Milk," by H. Faber.

"On the Quantity of Volatile Acids Present in Butter Fat," by Dr. Vieth.

"On the Amount of Volatile Acids in Butter Substitutes," by Otto Hehner.

FAT EXTRACTION FROM MILK SOLIDS.

BY H. DROOP RICHMOND.

(*Read at Meeting, May,* 1889.)

THE estimation of fat in milk, simple though it seems at first sight, has perhaps given more trouble to analysts than anything else, and on it, consequently, a very large amount of original work has been done. Happily, now the subject is much better understood than it was, and by the improved methods now in use something like concordance between separate analysts is to be reasonably expected. But there is still a want to be supplied, which is well expressed by Vieth (ANALYST, xii., 63 ; xiii., 49), where he says, "The difference between the percentages of fat extracted if the coil process is used . . . . and the fat extracted if the plaster process is used . . . . are smallest with skim milks, and increase with the increasing percentage of fat ; or, in other words, the differences are small where great difficulties for the complete extraction of fat exist, and comparatively large where no such difficulties exist." Vieth gives two tables illustra this: one in ANALYST, xii., 63, and the other comparing the results obtain Hehner and myself in working out our formula (ANALYST, xiii., 49). A third table ing the same thing is found in ANALYST, xi., 62 ; and a glance at the diagram to our paper (ANALYST, xiii., 24) shows it in a graphic form.

To explain this fact I undertook this investigation. I only propose to consider here the methods of extraction of fat from milk solids, and shall not, therefore, refer to such methods as Soxhlet's areometric process, etc.

The methods of fat extraction from milk solids are many, among them being:—

1. Wanklyn's direct extraction, and modifications by Carter Bell, Waller, etc.
2. Evaporation on Sea Sand.
3. „ „ Asbestos. Babcock (*Dept. Agric. Rept.*, 13, pt. I., 81).
4. „ „ Asbestos Cloth or Paper. Wiley (*Foods and Food Adulterations*, pt. I., p. 81), also used by Johnstone (ANALYST, xii., 234).
5. Evaporation on Plaster of Paris. Soxhlet.
6. „ „ Pumice, with grinding to powder. Storch (ANALYST, xii., 6).
7. „ „ Copper Sulphate. Morse, Piggott, and Burton (ANALYST, xiii., 18).
8. Evaporation on Sponge. Duclaux (*Le Lait*, p. 176).
9. „ „ Lint. Abrahams (ANALYST, ix., 20).
10. „ „ Paper. Adams (ANALYST, x., 46).
11. „ „ Powdered Glass. Baynes.
12. „ „ Kieselguhr.
13. „ „ Wood Fibre. Gantter (*Z. f. Anal. Chem.*)
14. Extraction from Milk Solids in pasty condition. J. Bell (*Foods*, vol. ii., and ANALYST, viii., 141).
15. Extraction from precipitated Casein.

It was at once seen that the investigation of all these methods would serve no useful purpose. It may be laid down as an axiom of milk analysis, that only methods which extract the whole of the fat are applicable, and no other should be tolerated as an exact estimation. The following published statements were found to assist in choosing suitable methods for investigation:—

1. Wanklyn's method and modifications are now universally admitted to be faulty.

2. By the use of sea-sand, Schrott and Hengold find results very nearly the same as Soxhlet's plaster and areometric methods; other experimenters find rather less.

3. Kent and Grothe (ANALYST, x., 106) find that Babcock's method gives only slightly more than Wanklyn's, and ·2—·3 less than Soxhlet's.

4. Wiley has discarded his asbestos paper method on account of it not giving results as high as Soxhlet's areometric method; Johnstone has published figures (ANALYST, xii., 234) showing that results obtained by asbestos cloth fall appreciably below those given by Fleischmann's formula.

5. Soxhlet's method, which is the usual method of fat estimation on the Continent, gives very concordant results, which are higher than any of the preceding.

6. Storch's method gives on an average ·27 higher than Soxhlet's (ANALYST, xiii., 6; *Versuch. Stat.*, 1888, p. 316).

7, 8, 9, 11, 13, and 15. No comparative figures published for any of these methods.

10. Adams' paper methods gives higher results than any other, except Storch's (ANALYST, x., 46; Baertling, *Rep. f. Anal. Chem.* 41 (1886), No. 31, 411, etc.).

12. Neither method nor figures yet published.

14. Bell's method has been examined by Wigner (ANALYST, viii., 211), and gives ·2—·3 more than Wanklyn's.

From these data I decided to submit to investigation the following methods:—

Soxhlet's plaster method.
Adam's paper ,,

d either Storch's pumice method or the Kieselguhr method, which are practically entical.

An objection has been urged against Adams' method on the ground that paper ntains a substance which is soluble in ether, but difficult to extract; thus Vieth NALYST, x., 86) found that one coil gave, after soaking over night in ether, 157 grs., and on a second extraction ·0072 grs. more; and Faber (ANALYST, xii., 8); ows that in one case a first extraction gave ·023 grs., and a second ·010 grs. The structions of the Milk Committee of the Society of Public Analysts (ANALYST, x., 217) e to previously extract the coils with ether, but nothing is said about there being any fficulty in extracting them completely, it being implied that twelve siphonings are fficient. In a former paper, in which Hehner and I used this method for working out a rmula, we stated that we did not previously extract our coils, but made a subtraction ·012 grs., that being the average amount extracted by blank experiments. Having w another batch of paper, the usual blank determinations were made, twenty-eight ils giving on an average ·0102 grs. each, giving

a probable error for one coil of ·0006 grs.
and a maximum error ,, ,, of ·0025 grs.

wo coils were submitted to further extraction, with the following results, each being tracted for 1½ hours at a time:—

1st Ext.	2nd Ext.	3rd Ext.	4th Ext.	5th Ext.	6th Ext.	7th Ext.
grs.	grs.	grs.	grs.	grs.	grs.	grs.
·0102	·0086	·0076	·0074	·0012	——	——
·0127	·0025	·0041	·0032	·0030	·0020	·0020

ving a total of ·0350 grs. and ·0295 grs. respectively. These results showed that hough the subtraction of certain quantity found by blank experiment might do for ery-day work, yet for a scientific investigation it is absolutely inadmissible. I tried e use of various other solvents; neither benzene, petroleum ether, or carbon disulphide owed any advantage over ether, but alcohol completely extracted the papers. As it s been stated that soap is used in the manufacture of the paper, I also tried the effect acid, either by soaking the coils in dilute acetic acid, allowing to dry and extracting th ether, or by mixing a small quantity of glacial acetic acid with the ether or alcohol ed for extraction, and found that in every case the paper was completely extracted. found that the most convenient and expeditious method of extracting papers was to e acid alcohol, *i.e.*, alcohol containing 10 per cent of acetic acid, and extract in a large xhlet apparatus for three or four hours. Paper extracted in this way yielded to her after about six hours' extraction a very small quantity of extract, consisting chiefly fibres, amounting to perhaps one milligram., which if necessary may be estimated and btracted, but which is practically negligible. After the completion of this stage of e research, my attention was drawn to a paper by Professor Soxhlet (*Molkerei Zeitung*, , 439), who had come to the same conclusions, and as the outcome of his work, essrs. Schleicher and Schüll are placing on the market a fat-free paper, of which their ents in England, Messrs. Townson and Mercer, 89, Bishopsgate Street, E.C., hold a ck.

To prevent an objection similar to that urged against the paper process being brought forward against the plaster or Kieselguhr processes, I discarded the use of paper to wrap the cartridges in, and substituted wide test-tubes, having a hole at the bottom, and containing a wad of cotton wool which had been previously extracted with ether, until it gave no further extract. The Kieselguhr was ignited before use. The plaster or Kieselguhr, after being evaporated, was ground in the basin in which it had been dried, and *not, as is usually done, simply crushed to a coarse powder ;* in many cases it was, after being extracted, ground a second time. Tho extractions in all cases lasted *at least three hours,* and in most cases more, as it was found that less time did not suffice, especially in the plaster extractions. For the sake of comparison, a series of results with unextracted coils were made, a correction of ·010 grs. being used, and the extraction being prolonged for only 1½ hours (our usual custom hitherto).

The fine and repeated grinding and long extraction are essential points of Storch's method, and should always be adopted if very exact results are wanted.

The following are the results obtained :—

No.	G.	$\frac{G}{D}$	T.	Ad. Ext.	Ad. Un.	P.	K.	Calc.	
								New F.	Old F.
1	32·3	31·3	11·62	2·89	2·95	2·85	*2·94	2·90	2·93
2	33·1	32·0	12·41	3·30	3·40	3·25	*3·31	3·41	3·43
3	29·6	28·7	11·05	2·81	2·91	2·76	*2·86	3·00	3·03
4	32·1	31·1	12·99	4·00	4·10	3·85	—	4·11	4·14
5	32·0	31·0	12·90	4·02	4·15	3·96	—	4·06	4·09
6	31·5	30·5	12·57	3·78	3·89	3·74	*3·85	3·90	3·92
7	28·2	27·4	15·08	6.77	6·91	6·73	—	6·73	6·78
8	31·2	30·3	11·07	2·66	2·81	2·66	—	2·65	2·69
9	27·1	26·4	16·80	8·37	8·47	8·35	—	8·43	8·49
10	30·2	29·3	11·82	3·45	3·49	3·41	—	3·51	3·56
11	29·7	28·8	13·92	5·45	5·54	5·41	—	5·43	5·47
12	31·1	30·2	11·10	2·75	2·88	2·70	—	2·70	2·74
13	31·2	30·3	11·29	2·77	2·89	2·77	—	2·84	2·89
14	30·8	29·9	12·57	3·94	4·49	—	—	4·03	4·07
15	31·8	30·8	12·06	3·35	3·54	—	—	3·38	3·42
16	30·3	29·4	10·52	2·41	2·62	2·40	*2·49	2·38	2·42
17	30·7	29·8	12·49	4·03	4·22	—	—	3·98	4·03
18	31·6	30·6	12·24	3·61	3·78	—	—	3·58	3·62
19	29·8	28·9	11·98	3·80	4·00	—	—	3·74	3·78
20	31·5	30·5	10·35	1·99	2·10	1·96	*2·02	2·00	2·03
21	32·8	31·8	12·69	3·86	—	3·80	*3·86	3·70	3·73
22	32·0	31·0	13·61	4·75	—	*4·75	*4·79	4·67	4.69
23	33·8	32·7	9·21	·54	—	* ·54	* ·60	·52	·53
24	35·4	34·2	9·41	—	—	* ·46	* ·44	·36	·37
25	35·6	34·4	9·29	—	—	* ·26	* ·27	·21	·22
26	35·3	34·1	9·37	·26	—	—	* ·26	·34	·35
27	32·5	31·5	11·47	2·68	—	—	*2·71	2·71	2·76
28	35·6	34·4	9·71	·42	—	—	* ·45	·56	·58
29	33·9	32·8	10·08	1·13	—	—	*1·13	1·24	1·26
30	—	—	—	·35	—	—	* ·35	—	—
31	—	—	—	—	—	* ·40	* ·44	—	—
32	—	—	—	—	—	* ·20	* ·21	—	—
33	—	—	13·87	5·00	5·07	4·96	†4·95	—	—
34	35·4	34·2	9·28	·18	·15 ·28	† ·10	† ·12	·25	·25

* *Re-ground* and extracted a second time. † Extracted 1½ hrs. only.

1-20 were extracted for about three hours, the others 5—6 hours.

These results lead to the following conclusions :—

1. If a sufficiently long time is given for the extraction, and if the plaster and ieselguhr are ground to a fine enough powder, there is, practically speaking, no differ- ce betwen the paper, plaster, or Kieselguhr methods.

2. The use of unextracted coils induces an error which is by no means constant rying from + ·06 to + ·55. The observation that acids rendered a larger portion of the tractive matter of the coils soluble than would otherwise have dissolved led me to the nclusion that the free acid in milk (always present) increased the amount extracted om the coil itself, and this was further shown by the following experiment :—A skim ilk, when fresh, gave as a mean of three results ·15 per cent. fat. A day or two after ards, when beginning to turn sour, the fat had increased to ·28 per cent., the correction ·010 grs. being made for each coil.

I certainly recommend that the instructions of the Milk Committee should be odified, and should state that the coils should be extracted with acid alcohol, and that t least three hours or more extraction should be allowed.

As I have shown that the paper method, as Hehner and I worked it (and, we elieve, as it was worked by many others), was open to objection, I deemed it advis- ble to see how my results agreed with the formula we had previously worked out, and hich was based upon that method.

A discussion of the various formulæ which have been proposed will not be out of lace here.

Behrend and Morgen (*J. f. Landw*, xxvii., 250) were the first to point out the act relation between sp. gr. fat and total solids, and published a table. In the same ear, 1879, Clausnitzer and Mayer (*Forchngen auf dem Gebeite*, ii., 265) gave a ormula, constructed from one sample of milk, which was analysed, allowed to stand during he night, and the skim milk again analysed. From this they found that 1 per cent. at lowered the gravity ·001, while 1 per cent of solids not fat raised it ·00375. Hehner ANALYST, viii., p. 129), from the result of 22 analyses of milk, compiled a formula to present Wanklyn's method ; he took the gravity of fat to be ·9275, and assumed that per cent decreased the gravity (water = 1,000) by ·725, while each per cent. of solids ot fat raised the gravity by 3·6. This assumption is not absolutely correct, though the rror introduced is well within the limits of experimental error. Fleischmann and orgen (*J. f. Landw*, xxx., 293) constructed a formula to represent the plaster process. hey assumed that each per cent. by volume (*i.e.*, gram. per 100 c.c.) of fat lowered the ravity by a constant factor ; while each per cent. by volume of solids not fat raised by a constant factor. They took the gravity of fat at 15·5 to be ·94. From a ries of experiments, Fleischmann found the gravity of butter fat to average ·93, d consequently altered his formula in accordance with this fact. Hehner and myself st year published a formula for the Adams' process, in which we made the same sumption as in Hehner's original formula, but obtained our constants entirely from our servations. None of these formulæ are absolutely free from objection, as they either ntain an assumption that is not strictly correct, or the gravity of the fat is assumed be the same in milk as it is in the solid state.

On account of this, and the fact that the method employed by Hehner and myself is now shown to be faulty, I have considered it desirable to obtain a new formula.

If we assume that $1+x$ is the gravity of a substance in solution, and that we have a solution containing y grs. in 100 c.c., the y grs. will occupy $\frac{y}{1+x}$ c.c., and as the total bulk is 100 c.c., the water occupies $100 - \frac{y}{1+x}$ c.c., and weighs $100 - \frac{y}{1+x}$ grs.

⁂ The specific gravity of solution is
$$\frac{100 - \frac{y}{1+x} + x}{100}$$
$$= \frac{100\,(1+x) - y + y + xy}{100\,(1+x)}$$
$$= 1 + \frac{xy}{100\,(1+x)}$$

⁂ 1 gram. in 100 c.c. raises the gravity by $\frac{x}{100\,(1+x)}$ which is a constant for each substance.

In a milk each gram. of fat in 100 c.c. lowers the gravity by a certain amount, (ϕ), while the solids not fat raise it by a certain amount (σ); and if $F =$ per cent. of fat, $F \times D$ (density) $=$ per cent. of fat by volume, and $S \times D =$ per cent. of SNF by volume, then $G = S \times D \times \sigma - F \times D \times \phi$, or $\frac{G}{D} = S\sigma - F\phi$; or as $S = T - F$: $\frac{G}{D} = (T - F)\sigma - F\phi$;

$$\text{or } \frac{G}{D} = T\sigma - F(\sigma + \phi)\text{; or } \left(\frac{\sigma+\phi}{\sigma}\right) - \frac{G}{D}\,\frac{1}{\sigma}\text{; or } 1 = \frac{F}{T}\left(\frac{\sigma+\phi}{\sigma}\right) - \frac{G}{D}\,\frac{1}{T\sigma},$$

and, by the same process as used in the paper by Hehner and myself, I calculate from my results the following formula: $T = 1{\cdot}17\,F - {\cdot}263\,\frac{G}{D}$, which gives the following values:—

$\phi = {\cdot}65$, sp. gr. of fat ·939
$\sigma = 3{\cdot}8$, sp. gr. of SNF 1·613.

In calculating this formula only the Kieselguhr results and the paper and plaster results of Nos. 22—29 have been used; the formula when applied to the paper results of Nos. 1—21 averages about ·02—·03 per cent. higher.

It is a curious fact that the sp. gr. of fat in milk should be ·939, which is distinctly higher than the observations of Blyth, Fleischmann, and others. This number is practically identical with the number calculated from the results obtained by Hehner and myself, and with that used by Fleischmann and Morgen. With regard to this it is worthy of note that Besana, from an extended series of analyses by Soxhlet's method, found that Fleischmann and Morgen's old formula expressed the results better than Fleischmann's new one; an examination of the results published in Fleischmann's paper (*J. f. Landw*, xxxiii., 251.) shows the same thing; and Vieth practically confirms this in a paper where he states that with skim milks from 141 analyses he finds there is an average difference of + ·04, which is about the difference between the two formulæ. It is, therefore, a remarkable fact that butter-fat has a higher gravity in milk (when presumably liquid) than it has when solid. Taking the gravity of milk-sugar to be 1·65—a number ned by many observers—of casein, etc. at 1·31 (Hehner), and ash at 3·0 (Dupré), and roportions of these constituents in milk at 6 : 5 : 1 (Vieth), the gravity of SNF d be

$$\begin{array}{lll} 1{\cdot}65 \times 6 & & 9{\cdot}90 \\ 1{\cdot}31 \times 5 & & 6{\cdot}55 \\ 3{\cdot}0 \ \ \times 1 & & 3{\cdot}0 \\ \hline & & 19{\cdot}45 \div 12 = 1.62 \end{array}$$

h is nearly identical with that given above.

When working out our formula, Hehner and myself attributed to lower results ned with skim milk to the fact that a change in composition (due to formation of ates) took place as less and less fat was present, or as the amount of water increased. certain if this was so, a sample of skim milk was taken and diluted with weighed tities of water, the mixtures allowed to stand several hours, and the density taken. density of the mixtures was calculated from the density of the original milk, and amount of water added, with the result that the two were identical within experial error.

Percentage, by Milk.	Volume of Water.	Observed Sp. Gr.	Calculated Sp. Gr.
100	—	1·03544	—
94·4	5·6	1·03343	1·03345
89·3	10·7	1·03170	1·03165
83·3	16·7	1·02950	1·02952
79·8	20·2	1·02829	1·02828
68·8	31·2	1·02439	1.02438

In the table I have given in the two last columns, the numbers calculated by the formula, and Hehner's and my old formula respectively, it will be seen that the ence between them is very small, and that for all practical purposes they are iden-

By permission of the directors of the Dairy Supply Co., Mr. Faber has applied ew formula to over 200 analyses made by him in 1885 in the laboratory of that any by Storch's method, with the result of almost absolute agreement, there being verage difference of ·02 per cent., with maximum differences of + ·34 and — ·26 ctively, showing that this method, which has been in use by Storch and other Scanian chemists for several years, gives complete extraction.

As the total solids are used for the calculation of the fat, it is, of course, important they should be accurately estimated. The usual mode of doing this is to evaporate of milk to dryness in a small platinum, glass, or porcelain basin, these being used riminately. With a view of shortening the process Gerber and Radenhausen in proposed the use of acetic acid (*Vorschläge zur einer einheittleihr Untersuchungsmethod ilch* 7), but rejected it ultimately on account of the approximate character of the s, it being impossible to remove all the acetic acid, and at the same time to prevent e in the milk residue. Sea-sand and other powders have been used, Duclaux *ait*, 176) uses sponge, Babcock asbestos, and Adams paper, all with the idea of ding the residue out in a fine condition, so that perfect desiccation may take place. performing duplicate experiments, I was struck by the fact that when the milk in a small basin the results were lower, and the residues darkened more than when ger basin was used, and this was especially the case when the small basin was of porcelain. A series of experiments was made to ascertain the influence of mass on the estimation, by comparing the results obtained with from 1 – 1·5 grs. of milk in a large basin and 5 grs. in a small basin. The results are as follows:—

T. S. 5 grs.	T. S. 1 – 1·5 grs.	Diff.
10·57	10·62	+ ·05
12·33	12·41	+ ·08
10·89	11·05	+ ·16
12·79	12·99	+ ·20
12·79	12·90	+ ·11
12·53	12·57	+ ·04
14·97	15·08	+ ·11
11·06	11·07	+ ·01
16·69	16·80	+ ·11
11·70	11·82	+ ·12
13·77	13·92	+ ·15
11·05	11·10	+ ·05
11·22	11·29	+ ·07
10·25	10·35	+ ·10
12·60	12·69	+ ·09
13·42	13·61	+ ·19

In every case the results obtained with small quantities in large basins were higher than the large quantities in small basins, and the residues in the former case were quite white, while the latter were more or less brown. Using small quantities only, from one to one and a-half hour's drying is requisite, although longer drying produces no change in weight, one, No. 2 in the list, having been allowed to remain in for six hours, showing not the slightest change; using the larger quantity constancy of weight is not obtainable. I am unable to account satisfactorily for this, but it seems to be connected with the rate of drying, the bulk of the water being evaporated in two or three minutes in in the case of the small quantities, while it takes sometimes half an hour with the larger quantities. There is not the slightest difficulty in weighing out a small quantity of milk on a quick weighing balance, the loss by evaporation rarely being sufficient to influence the second place of decimals, and agreement between duplicates being extremely small (not exceeding ·05 per cent.). Wiley has made the same observation, and recommends in his book (*Foods and Food Adulteration, part i.*) either to dry about 2 grs. in a wide platinum basin or to dry 5 grs. on asbestos (*Babcock's method*). He finds that these two methods give identical results, which are higher than the usual method. Mr. Faber (private communication) informs me that he has dried 10 grs. of milk on 10 grs. of pumice, and finds that there is no loss of weight on leaving this overnight in the water oven. It is seen that there is a tendency to under-estimate the total solids, and, therefore, to over-estimate the water in a milk; this I mention because it has been used as an argument in favour of Soxhlet's method of fat extraction, that by its use complete analyses added up to about 100 per cent., Vieth (*Anal. xii.* 59) stating that his analyses add up to 100·15 on the average. I do not, however, propose to discuss this question here, beyond mentioning that Wiley (*Foods, etc., pt. i.*, 115) gives 121 complete analyses of milk, of which the average is

Water.	Fat.	Proteids.	Sugar.	Ash.	Total.
87·22	4·07	2·73	4·90	·67	99·59

which is ·41 below 100 per cent. His estimations were made as follows:

Water: by drying 2 grs. in wide basin, or 5 grs. on asbestos.

Fat: Soxhlet's areometric process.

Proteids: nitrogen by either soda-lime or Kjeldahl, × 6·38. (N.B.—Wiley uses 6·25, but I have recalculated his figures).

Sugar: polarised (Wiley's figures corrected for volume of fat).

Ash: as usual.

THEORY OF FAT EXTRACTION.

The difference between Adams' method and Soxhlet's method having now been shown, to be the result of, on the one hand, extraction of something, not milk-fat, and on the other, incomplete extraction, it only remains to explain, if possible, the theory of the subject. If an ordinary milk is filtered through filter or blotting paper, we find the portion that passes through is almost entirely free from fat, it usually containing ·1–·2 per cent.; showing that by capillary attraction between the fibres of the paper the milk is almost entirely separated into two parts, one rich in fat, which remains on the surface of the paper, and the other poor in fat, which passes into the paper itself. Here are the conditions favourable for the complete extraction of the fat. Then again, the milk is spread out over an enormous surface, so that a very thin film is presented for extraction, but I consider that this condition is far less effective in promoting complete extraction than the first separation. In Soxhlet's process the milk is absorbed almost as a whole by the plaster of Paris, and the mass is dried together, so that there is by no means the separation that occurs in the Adams process. This mass is roughly broken up only, and consequently it is not surprising that complete extraction does not take place. With regard to the increasing difference with the increase of fat, the difference is only apparently an increasing one; for instance, say with a milk containing ·5 per cent. of fat, there is a difference of ·2 per cent., and with a milk containing 5 per cent. there is a difference of ·4 per cent. At first sight there appears to be an increasing difference, while in reality, in the first case 40 per cent. of the fat is left behind, while in the second, only 8 per cent. And this is by no means surprising when we consider that almost the whole of the fat is distributed equally through the plaster, and that there is a great tendency for the plaster, etc., to enclose some of the fat, and the enclosing action would be greater the less the quantity of fat, and consequently there would be a tendency for a larger proportion of the fat to be left behind, although on superficial observation the amount of fat left in appears smaller. It is but natural to expect that by fine grinding and regrinding, and long extraction the enclosed portions of the fat should be brought into contact with the ether and become dissolved. It might be brought forward as an argument that the fat by long extraction becomes oxidised and therefore increases in weight; but this is excessively unlikely, and seems to be contradicted by the results of Johnstone (*Anal. xi.* 22), and the figures obtained by the use of the Abbé refractometer (for the loan of which I am indebted to Mr. Faber). Fats extracted by—

Adams' method.	Soxhlet's (modified).	Kieselguhr process.
1·464	1·464	1·463
1·4655	1·462	1·465
1·463	1·463	1·462

These figures are all normal for butter-fat. When, however, fats are heated in contact with the air, the refractive index rises very perceptibly, thus a sample of butter-fat gave at first 1·463, after six hours in water-bath at 98°-99° C although the weight had not perceptibly changed, the refractive index was 1·467. I do not therefore think that that objection can be maintained; there was nothing extracted by ether from any of the materials used, the paper being extracted beforehand, the plaster giving nothing, and the Kieselguhr, having been ignited before use, also giving nothing.

In conclusion, I would recommend that extracted papers be always used for the Adams process, and that the extraction should be continued for 3-4 hours as a minimum; or that the Kieselguhr process should be used as an alternative; Kieselguhr presenting many advantages over plaster, it being of a porous nature, and easy to reduce to a fine powder, through which ether percolates with comparative ease; and that total solids be taken by drying 1-2 grs. of milk in a wide platinum basin on the water-bath for about an hour and a half, cooling and weighing; this on returning to the bath for another half hour, rarely shows more than one or two tenths of a milligram. difference.

On account of the practical identity of the new formula and the old one worked out by Hehner and myself, I do not give a table for facilitating calculation, the one published in our paper (*Anal. xiii.* 32), and also as a separate sheet, and the "milk scale" (*Anal. xiii.* 65) adequately serving that purpose.

I wish to express my thanks to Mr. Hehner, in whose laboratory this research was carried out, for the kind way in which he has advised and superintended the work, and to Dr. Vieth and Mr. Faber for the loan of instruments, books, etc., and for the way in which they have placed at my disposal a large amount of valuable information.

DISCUSSION.

MR. ALLEN said that Mr. Richmond had brought before them a large number of valuable results, which they would study at their leisure. It was a matter for great satisfaction that they had a young chemist amongst them who was not content simply with accumulating figures, but who also strove to ascertain how far they were due to avoidable experimental error, and how far to some error inherent in the process. Mr. Richmond's suggestion as to the presence of soapy matter in the paper was very plausible, but at the same time he (Mr. Allen) would like to know whether Mr. Richmond had succeeded in extracting any resinous or fatty matter from the paper. As to No. 14 experiment he should think that either the paper was full of soap or else the milk was.

With regard to the notes on the different factors for calculating fat, he thought that was a refinement which they scarcely required. He always used Hehner and Richmond's slide-rule, which usually gave satisfactory results, but after all the method was only one of a preliminary kind.

The questions of the size of the basins and so on were all eminently practical, and they were all indebted to Mr. Richmond for bringing the matter before them.

DR. VIETH said that as Mr. Richmond had alluded to him as being partially responsible that the paper was brought before the Society, he wished to say that he took with pleasure his full share of responsibility upon himself. He thought they had heard an extremely useful and important paper. The figures given bore out a fact which had never been shown before. They all knew that something could be extracted from paper, but here, for the first time, they had a large number of comparative experiments showing that a larger quantity of ether-extract was obtained when milk was dried up on paper extracted in the usual way, than in case the paper was extracted under certain precautions. The experiments answered a number of other questions, for instance, they proved that laster of Paris was after all a very good medium for drying milk previous to extraction. e thought the results with regard to the determination of the total solids were of lmost more importance than the fat results. He was very much struck to hear that hen a very small quantity of milk was dried in a large basin the results should come ut higher than when a larger quantity was dried in a small dish. The experiments ere most interesting and important, and opened up a new field for inquiries which were eally wanted in that direction. With regard to the figures quoted from Dr. Wiley's eport to the Commissioner of Agriculture, U.S., he must say that he considered the ercentage of proteids so extremely low that he had some doubt whether the results ould be correct. Personally he wished to thank Mr. Richmond for his valuable paper, hich cleared up some hitherto rather obscure points.

Mr. Stokes said that in the figures given of the three processes, if the at was below 4 per cent. or thereabouts, those methods always gave a lower esult than the calculation, whether by the new or the old table, whereas when ou come to fats higher than 4 per cent., you always extract more than the calculation hows. With regard to dishes, Mr. Stokes thought some people made a mistake when hey said "large"; and he illustrated this on the blackboard, showing that so-called large" dishes had only higher rims but exposed no larger surface of the fluid. He dvocated the use of flat-bottomed dishes.

Mr. Richmond in reply said that he had not yet collected sufficient of the extract om the coils, but hoped to be able shortly to give some information respecting its mposition; with regard to the formulæ he had brought forward a new one, more from e scientific point of view than from its having any practical advantage over the old one. r. Vieth had pointed out that the proteids in Wiley's complete analyses were very low, nd he agreed with him there, but did not consider that the factor for converting nitrogen to proteids (6·38) was one on which any doubt could be shown, it having been established by Hammarsten and others for both casein and albumen in milk; he considered ther that, if there were a deficiency, it was more to be explained by Atwater's recent ork on the soda-lime method, where he found that even experienced observers are often d into grave under-estimations. He did not consider that in plaster extraction a rich ilk after one extraction was under the same conditions as a poor milk, for it must be membered that in a rich milk the proportion of molecules of fat to molecules of diluent n this case plaster and other milk solids) was much greater than in a poor milk, and nsequently in the poor milk each molecule of fat was confined by many more molecules diluent than in the rich milk, and so would be less easy to extract, and the extraction some of the fat molecules in the rich milk could not make the others less easy to y marked degree. He had observed that with plaster the fat was extracted less readily an with either paper or Kieselguhr.

With regard to Mr. Stoke's objections to the formula, he thought that they arose om insufficient perusal of the results, as the formula had been calculated from the e results themselves by a method which could not give other than agreement all along e curve, and was convinced that Mr. Stokes would see it in the same light when he d the paper before him and could study it at his leisure; he need hardly say, however, at he must not expect formulæ to agree to ·012. As to size of basins, he called a large sin one which exposed a large surface of milk to be evaporated, and a small one in hich the opposite was the case. In reply to Dr. Voelcker he admitted that when milk awn from the cow was not acid, he believed that it had the property of seeming both id and alkaline at the same time, but by the time that it usually reaches the hands of e analyst it has become slightly but distinctly acid, due, no doubt, to the commencement of lactic fermentation.

[Note.—Since reading the paper I have examined the extract from the coils, and find it to consist iefly of the calcium salt of a resinous acid, containing also sodium and small quantities of aluminium. H. D. R.]

THE DISCRIMINATION OF THE VARIOUS SPECIES OF SACCHAROMYCETES.

By Walter J. Sykes, M.D.

(*Read at the Meeting, May,* 1889.)

This has been considered hitherto simply a matter of microscopic observation; Rees and others have given descriptions and figures of the various yeast plants, but it has been recently shown by Hansen that each species of yeast is capable of assuming, under varying conditions of cultivation, etc., nearly all the forms which had been assumed as properly belonging to the others. Thus a circular, or nearly circular form is associated with the Saccharomycetes cerevisiæ group, an oval one with the Ellipsoideus group, and an elongated or sausage-shape form with the Pastorian group; but as under certain circumstances Sacc. Cerevisiæ forms oval and spindle-shaped cells, and the others in their turn appear as circular cells, any absolute determination of their species by the microscope alone must naturally be futile. As will be seen further on, the ability to differentiate the various species of yeast is likely to become a matter of considerable importance, and one which the analyst ought to be prepared to undertake. Before going into the question of resolving yeast into its various species, it will be well to say a few words about yeast itself.

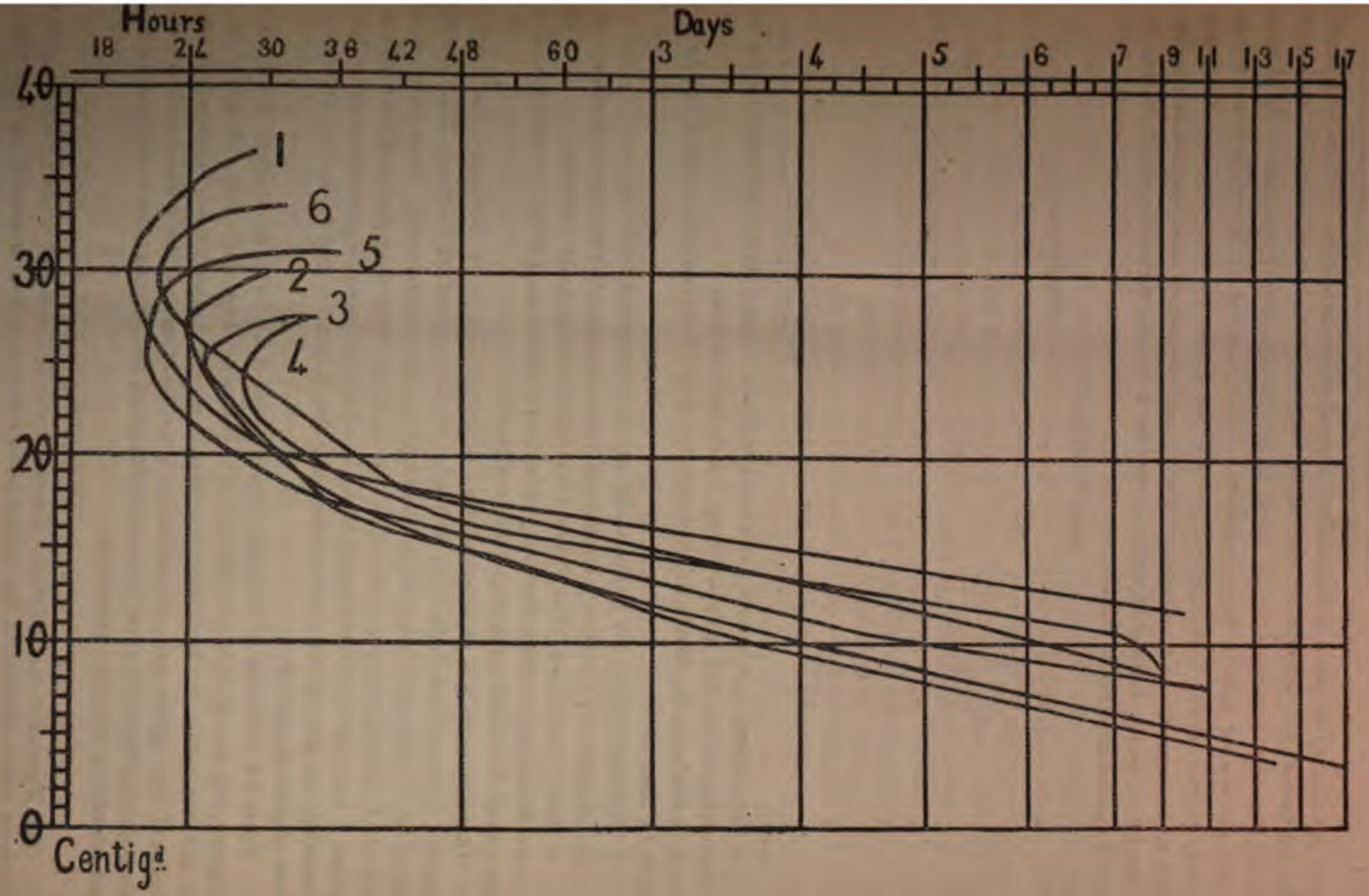
The true yeasts are simple unicellular plants belonging to the genus Fungi, and as they are capable of developing spores in cells called asci, they belong to the Ascogenous division of that genus. Yeast can reproduce itself under two distinctly different conditions; the ordinary every-day one, where, immersed in a liquid capable of undergoing fermentation, it rapidly increases by a process of budding, never in this case forming spores. The other condition was first brought into notice by Rees in 1869, who found that by suddenly depriving yeast of all saccharine food, and placing it on the surface of a slice of potato or other moist porous vegetable root, in a very thin layer, budding still went on for a short time, but eventually ceased, and in a number of the cells spores were formed. Other observers obtained the same results by cultivating yeast on blocks of plaster of Paris, kept moist; and this more convenient method is the one now universally employed for this purpose.

To Pasteur we owe our first definite knowledge of the relation of the organised ferments to the process of fermentation. He conclusively proved that fermentation only proceeds when the ferment grows and multiplies; he also taught us that such organised ferments as the acetic, lactic, butyric, etc., together with baccilli, bacteria, etc., frequently contaminated our brewing yeast, and were the causes of what are commonly known now as the diseases of beer, viz., acidity, instability, ropiness, etc. He also very clearly pointed out the way to get rid of these troublesome invaders, and in this way did inestimable service to the brewing industry. He, however, states that when you have thoroughly purified a yeast, so as to be entirely rid of all adventitious organisms, you may yet obtain a yeast which gives a beer of bad flavour. This fact, which Pasteur left unexplained, has been entirely cleared up by the brilliant work of Dr. Hansen, the Principal of the Physiological Laboratory at Carlsberg, Copenhagen. He found that yeast, as ordinarily used in breweries, is an indefinite mixture of yeast plants of various species and varieties; that the species and variety exercise a most marked influence on he flavour and properties of the finished beer, some causing a nauseous, bitter flavour thers producing an article which will not clarify, others an unstable article, etc. He roposed to introduce into practice, and has actually done so, yeasts grown from a single ll, and therefore consisting of one distinct species only. The advantages of this system re obvious; the brewer, provided he takes care to use good materials, can invariably ecure a beer of identical flavour and other properties. That this is not a mere matter f laboratory experiment is shown conclusively by the fact that these single cell yeasts ave been used since the year 1884 in the Old and New Carlsberg Breweries, which roduce annually over seven million gallons of lager and export beer, with the most arked success. In Denmark and Norway they are successfully used in all the largest reweries, to some extent in Sweden, Finland, Bohemia, and other parts of Austria, in witzerland, North Italy, Belgium, North America, Russia, Germany, and Holland, in sia and Australia. In all these breweries, with the exception of Baartz and Zoon, at otterdam, the system of low fermentation is adopted, *i.e.*, the type of yeast used is a ottom fermentation one, the fermentation being conducted at a temperature of 6 to 10° ent.; the yeast sinking to the bottom of the fermenting vessel. The Rotterdam rewery employs the high fermentation system, which is the one almost, if not in- ariably, used in this country. In this system a yeast is employed which, as fermenta- on proceeds, rises to the surface, the temperature of the fermentation being from 10 to 5° Cent. So far the high or top fermentation yeasts have been very little udied, but as the advantages to be obtained by employing single-cell yeasts are sure to recognised sooner or later in this country, a highly-interesting field of practical ob- rvation and research is opened out. The preparation of the small samples of the fferent varieties of single cell yeast will naturally fall within the province of the emist; their multiplication to quantities sufficient for practical purposes, will take ace in properly constructed apparatuses in the brewery.

The method devised at first by Hansen to obtain a sample from one cell was as llows: he diluted the yeast with water so that a measured quantity under the field of e microscope contained, say twenty cells. A quantity of this mixture, equal to that ntained in the microscope field, was added to 20 c.c. of water and well shaken up. It as now assumed that one c.c. of this mixture would contain a single yeast cell. This antity was next added to wort contained in a Pasteur or similar flask, and vigorously aken, the remaining 19 c.c. being treated in a similar manner. The flasks were amined in a few days, and if bubbles were seen proceeding from one point only, the periment was considered successful. If no sign of fermentation appeared, it was idence that the c.c. of the mixture introduced had not contained a yeast cell; if bbles arose from two or more points, that more than one yeast cell had been intro- uced, and naturally such flasks were rejected. This method had considerable elements of ance about it, and I only mention it as the one by which all Dr. Hansen's discoveries ere made, particularly as it has been hinted by some of his detractors that studies had their origin in Koch's gelatine plate cultivation process. This could not the case, because all Dr. Hansen's facts were published before the latter's gelatine ocess had been announced.

After the publication of Koch's method, Hansen adopted a modification of it, which leaves nothing to be desired in the way of obtaining cultures from a single yeast cell. He proceeds as follows: A sample of yeast is diluted as before with water until each microscopic field shows about twenty or thirty cells; a small drop of this mixture is now mixed thoroughly with 20 c.c. of ordinary beer wort containing 10 per cent. of gelatine. In all these processes it is almost unnecessary to state to my present audience that apparatus and fluids used must all be thoroughly sterilised. The gelatinized wort must never be allowed to reach a higher temperature than 25° Cent. at the time of the addition of the yeast cells or afterwards, for obvious reasons. A drop of this is spread in an even layer on an ordinary microscopic cover glass, placed film side downwards, on a small moisture chamber, containing a small quantity of water. The whole is placed under the microscope, and if the experiment has been successful, some half-dozen isolated yeast cells will be observed in different parts of the field, each of which is destined to form a colony. Those sufficiently apart are chosen from the rest, and a ring marked round them by a small apparatus which screws into the place of the object glass. A number is affixed to each circle with a fine pen, and the figure of each cell and its number noted down in the memorandum book. The moist chamber and its contents are placed in a warm place for 24 hours and further examined. Each cell will now be found to have formed a small colony; all the marked colonies are carefully examined, to see they are not likely to coalesce with adjacent ones, and that they are perfectly circular, great stress being laid upon this latter point by Dr. Hansen. After seeing that all is going on right, the moist chamber is allowed to stay another 48 or 72 hours, when the colonies will be found so large as to be easily seen by the naked eye. They are each in turn picked off the gelatine film on small pieces of platinum wire, which are dropped, colony and wire, into separate cultivation flasks containing wort. Fermentation and multiplication of yeast cells now commences, and all that is necessary is the careful periodical changing of the wort, so as to avoid outside contamination. By this means it is possible to increase the growth of yeast to any amount. The next question was to establish a method of differentiating the species with certainty, and this problem our indefatigable observer, Dr. Hansen likewise solved satisfactorily. By observing the occurrence of spore formation at different temperatures, he discovered that each species only developed spores between certain temperatures, and that at temperatures between these limits each species had its own relation to time and temperature. By taking the times of development as abscissæ, and the temperatures as ordinates, a curve may be generated for each yeast.

The accompanying diagram represents such a series of curves for six yeasts. For the abscissæ I have used a logarithmic scale of numbers, by which the initial curves are seen more plainly than if the ordinary numerical scale had been used. Spore formation is best observed in the following manner: A small truncated cone of plaster of Paris is moulded, the upper surface of which must be perfectly flat and smooth. On this surface, the yeast to be observed must be spread in a thin layer, the cone is placed base downwards in a vessel, and water poured in until it reaches halfway up the side of the cone. The whole is loosely covered, so as to admit of free aeration, this being absolutely necessary for spore formation, and kept at an even temperature. Small samples are taken off the surface of the cone from time to time and examined under the micro-


Hours
Days
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scope; the time to be noted is the earliest sign of spore formation. It has been found that only young and vigorous cells sporulate; care must be taken, therefore, to secure these by growing the sample for 24 hours in good, well-aerated wort, carefully pouring this off, and adding a fresh supply of wort. After another 24 hours the wort may be carefully poured off the thick layer of yeast at the bottom of the vessel, and the latter spread on the plaster block as described above. In this manner Hansen has differentiated six species of yeast, Saccharomycetes Cerevisiæ, a top fermentation yeast, having its times of spore formation as follows:—

Sacc. Cerevisiæ I.

37·5	No spore formation.
36·37	29 hours.
35	25 ,,
33·5	23 ,,
30	20 ,,
25	23 ,,
23	27 ,,
17·5	50 ,,
16 5	65 ,,
11-12	10 days.
9	No development.

S. Pastorianus I., a bottom fermentation yeast, which frequently occurs in the air of fermenting rooms, and gives a strong, bitter taste to beer.

S. Pastorianus II., feeble top fermentation yeast, does not seem to give rise to any disease in beer.

S. Pastorianus III., top fermentation yeast, causes yeast turbidity.

S. Ellipsoideus I., bottom fermentation yeast, found on the surface of grapes.

S. Ellipsoideus II., bottom fermentation yeast, causes great turbidity.

As the whole nomenclature of yeasts is at present in a state of transition, these names must be looked upon as merely provisional.

In addition to the before-mentioned discoveries in spore formation, Dr. Hansen observed some curious facts with reference to the formation of yeast films on the surface of a yeast cultivation when fermentation had quite ceased. Each yeast seems capable of forming a film, which appears at a longer or shorter period of time for each species. Temperature effects a similar acceleration or retardation, as in the case of spore formation, and consequently the conditions of film formation may be used to corroborate those of spore formation.

From the foregoing we conclude that diseases of beer are not only caused by false ferments, but also by saccharomycetes; that it is possible to distinguish the useful from the noxious species of the latter; and that it is easy to prepare a yeast which shall consist of one species only, thus affording the brewer an exactitude and certainty in his fermentation results he never possessed before, and at the same time opening out a wide and interesting field for the analyst.

DISCUSSION.

The PRESIDENT said he would like to know if Dr. Sykes could explain definitely the cause which determined the matter as to whether a yeast became a top or a bottom yeast. Was it due to an entirely different kind of cell, or was it due to the circumstances under which the cell was placed? Had Hansen's researches confirmed the belief a yeast became top yeast, when by its treatment the growth was encouraged and became vigorous, or on the other hand bottom yeast when its growth was checked. Of course it was understood that top yeast fermentation took place at a higher temperature, and that in proportion the growth of the yeast plant was more rapid, and thus gas was formed more abundantly, which, by adhering to the yeast cells, rendered them buoyant and carried them to the top, so forming top yeast; but with the lower temperature and less vigorous growth and less gas formation, bottom yeast resulted.

With respect to the modification which Hansen has shown by his experiments to be capabe of cultivation, is it not true that these varieties are very unstable, and readily revert to the ordinary type?

He, the speaker, would like also to ask Dr. Sykes whether he had any knowledge of the enduring qualities of the ascospore formation, as compared with the ordinary method of reproduction. From a biological as well as a sanitary point of view, the matter was of great interest to him.

Mr. Richmond, bearing in mind that Dr. Sykes had stated that Sach. Pasteureanis I. gave bitter taste to beer, would like to know if the substance giving this taste was precipitated by acetate of lead, otherwise it might interfere with the detection of foreign bitters.

Mr. Cassal asked if they were to understand that one sort was absolutely incapable of being converted into any other sort? He would like to know if any experiments had been made with a cell as to its capability of altering its original constitution as evidenced, for example, by the appearance of abnormal products, such as the bitter substance which had been alluded to. It had been asserted in regard to micro-organisms obtained from "pure cultivations," that they were incapable of change in this respect, but he greatly doubted whether there was sufficient evidence to warrant such a statement.

In reply to the President's questions Dr. Sykes said the attempt to transform one species of yeast into another had been tried in every conceivable way by Hansen, and found impossible; he considers that the species are as distinct and definite as that of any of the higher fungi. You may cultivate high yeast at a low temperature, but though it sinks like a low yeast, fermentation proceeds much more slowly than in the case of a bottom fermentation yeast; if placed in its normal fermentation temperature, and especially if well aerated, it quickly resumes its former properties. In making a gelatine cultivation of a yeast known to be of one species, you might find two colonies of cells of entirely different shape, as, for instance, one oval, the other sausage-shaped. If these were introduced into two flasks, and a cultivation started, though the whole cells formed at first would be of the same shape as their progenitors, yet sooner or later this speciality of form would entirely disappear. With reference to the spores of yeast they are remarkably persistent. About a year ago he had found that they existed in large quantities on malt, which is generally exposed to a temperature of 200° to 220° F. for several hours during the final stage of drying on the kiln.

In reply to Mr. Richmond's question, he did not know whether the bitter generated by S. Pastoriensis I. was precipitable by acetate of lead or not.

In reply to Mr. Cassal, Dr. Sykes saw that whatever changes took place in the shape, etc., of the cells by different methods of cultivation, their chemical properties remain unaltered; a Cerevisiæ would always produce a good flavoured beer, a Pastoriensis I. would invariably give one of bad flavour.

(*Conclusion of the Society's Proceedings.*)

ON THE ESTIMATION OF STARCH.

By Prof. Dr. Alex. von Asbóth.

Having to attend to many professional duties, I have been prevented from sooner completing my investigations on the estimation of starch in cereals. Last July (see Analyst, 1888) I proved by several experiments that in working with pure starches, the results obtained by Lintner's process and my baryta process were concordant. I therefore concluded that equally concordant results would also be obtained in dealing with cereals, but I was much mistaken. I experienced the same thing as Monheim, who found in the same sample of wheat 69·06 per cent. of starch by my process, and only 62·08 by Lintner's. I then began to think whether cereals might not contain other baryta absorbing bodies, and at first suspected the proteids, but afterwards found these not to interfere. After many experiments I found the enemy to be fatty matter.

It seems that the fats contained in cereals contain free fatty acids, or these acids are in such loose combination with glycerin, that they are easily saponified by the baryta water. This being so, it was easy enough, so to modify the process, as to render it fit for the analysis of cereals. The flour must be first extracted with ether, which I do as follows:—Ten grammes of the substance are put into a filter paper cartridge and exhausted with ether in the Soxhlet in the usual manner. The solution is evaporated to dryness and the fat weighed. The contents of the cartridge are now put on filter paper and exposed for about an hour, so as to let every trace of ether go off. The flour is then thoroughly mixed in a porcelain mortar, and is then ready for analysis. A few grammes are used for the estimation of moisture, and 1 or 1·8 grm. is used for the treatment with baryta. This modification of the process involves a little calculation, of which I give an example. From a sample of finely ground wheat I took 10·0235 grms. for the estimation of the fat. Obtained ·1785 grm. fat amounting to 1·78 per cent. The moisture of this sample dried at 110° C. came to 13·29 per cent. From the *extracted* flour I weighed 1·511 grm. and, after gelatinising it as directed, added 50 c.c. of baryta water and made up to the mark with 45 per cent. alcohol. 10 c.c. baryta water = 29·53 c.c. $\frac{N}{10}$ hydrochloric acid[*]; 50 c.c. of filtrate = 23·04 c.c. acid = 69·58 per cent starch. The moisture of the extracted flour being only 11 per cent., we have :—

$$a.\ (100-11) : 69{\cdot}58 = 100 : x \qquad x = 71{\cdot}17.$$

$$b.\ 100 : 78{\cdot}17 = (100 - 13{\cdot}29 + 1{\cdot}78) : x$$

$$x \text{ (or the starch in original sample)} = 66{\cdot}37 \text{ per cent.}$$

I now also analysed the sample by Lintner's process, but could not get the results to agree, but as they agreed when dealing with pure starches, I began to suspect Lintner's process when applied to cereals. I therefore tested the residue which was left in the high pressure flask. On moistening with iodine, a blue colour was developed, and that even after heating for six hours at 140—145° C. As however cellulose also gives sometimes a blue with iodine, this test was not considered sufficient, and I therefore heated the residue with perfectly pure diastase for 12-24 hours at 65·70° C. The filtrate trongly reduced the alkaline copper solution. I also have made experiments to quanti- atively estimate the starch in the residue and operated as follows:—The precipitate rhich had collected on the glass wool was rinsed into a flask, and mixed with a solution f ·02 grm. of diastase. The whole was then heated for 24 hours at 65-70° C. in a rater bath. I then filtered off and diluted up to 100 c.c., then added 10 c.c. hydro- hloric acid and heated one hour in boiling water. The liquid was next neutralised rith barium carbonate, and evaporated to dryness in the water bath. The residue was hen exhausted with alcohol of 90 per cent., filtered off, again evaporated, and the sugar nally estimated with Fehling's solution. The result could not be right, because it is ot to be taken for granted that all the starch had dissolved, and the inversion process was one in too short a time, but I did not wish to heat longer so as to prevent decomposi- ion of the diastase by the acid, which would yield sugar. However, I still obtained rom the residue ·83 per cent. of starch, calculated on original substance.

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[*] I now prefer to first use $\frac{N}{10}$ acid, and when nearly enough of this has been added, I use $\frac{N}{100}$ acid With care ·005 c.c. of a $\frac{N}{10}$ acid may thus be read off.

(*To be continued.*)

PEPPER ADULTERATION.

By F. Wallis Stoddart.

HE form of pepper adulteration described in a note appearing in an earlier number of his journal (Analyst, xiv., 37), is still largely employed, but the "make-weight" now rincipally introduced appears to be steatite in fine powder, which is added to the pepper o the extent of 10—15 per cent.

The proportion of chromate of lead remains about the same, and can, I find, be letected more readily in the ash than in the chloroform residue; for the latter not only ontains a good deal of pepper-starch, which interferes with the boiling, but also ap- arently some substance which renders the tests for chromic acid uncertain. One or wo grammes of pepper yields excellent results when treated in this way.

The feebleness of the present Act is well illustrated by the powerlessness of the uthorities to deal with this fraud; for, though the manufacturers are perfectly well nown, and numerous convictions amongst their customers have taken place, as they do ot reside in Bristol the local authorities decide they can do nothing with them; and, s in each case they openly offer to pay all fines and expenses, and, where necessary, even o salve the wounded reputation by a small honorarium, they are not likely to lack usiness.

LAW NOTES.

Battle of the Pharmacopœias.—At the Bow Street police-court, on May 27th, Charles erdinand Keele, chemist, of Tottenham Court Road, was summoned for selling sal volatile not repared according to the requirements of the British Pharmacopœia.—Mr. Smith appeared to support he summons; Mr. Forrest Fulton for the defendant.—On the 6th April, six ounces of sal volatile were ought at the defendant's shop for 3s. It was analysed by Dr. Redwood and found to contain less lcohol and less spirits of ammonia than were required by the Pharmacopœia Britannica. Some ques- ion arose as to the Act under which the summons was taken out, the Food and Drugs Act, and not he Pharmacy Act.—Mr. Fulton said it was matter of common knowledge that the new Act now efore Parliament would incorporate the two Acts. His client was not a "compounder" of this drug, ut simply a retail vendor. What he sold was also what was described as sal volatile, although under nother name in the Pharmacopœia Londonensis.—Mr. Vaughan asked to have some professional vidence on which to go, and Dr. Redwood was then called. He was asked by Mr. Vaughan what sal olatile should be, and replied, "A solution, partly aqueous and partly spirituous, of carbonate of ammonia, with certain essential oils of nutmeg, cinnamon, and lemon, thereby constituting a stimulating alkaline solution."—Mr. Vaughan: But in those ingredients I don't notice where the alcohol comes in.—The witness: Estimating it as a rectified spirit, there should be one-third.—Mr Smith: But as a medicine, what is the most important?——Witness: The ammonia.—Mr. Vaughan: But what is the alcohol for?—Witness: To keep the oils in solution. In the present sample there was less than half what there should have been of alcohol.—Mr. Vaughan: What is the test?—Witness: The great test is the power of neutralising oxalic acid.—Cross-examined by Mr. Fulton: Sal volatile was first used in medicine in 1720. It dropped out of use until 1885. It was mentioned under another name in the last edition of the Pharmacopœia Londonensis of 1851. This sample was of the same specific gravity as the preparation mentioned in the London Pharmacopœia, and was practically the same preparation, but in witness's opinion the British Pharmacopœia entirely superseded all others, and all articles ordered in medicine should be prepared in accordance with its requirements. For instance, hydrocyanic acid (prussic acid) was double as strong in the London as in the British Pharmacœpia, and any chemist making up a prescription written with the intention of having the medicine made up according to the latter, might cause very grave results if he went by the former pharmacopœia.—The case was adjourned until June 1st, when Mr. Vaughan gave judgment as follows:—The summons against the defendant was for selling sal volatile which was deficient in alcohol and alkaline salt according to the acceptation of sal volatile as an article of commerce. Professor Redwood, both in his analysis and in the evidence he had given in this Court, distinctly stated that the article sold did not come up to the requirements of the British Pharmacopœia by one-half. This was not denied, and the next point to be considered was whether it was lawful to sell drugs prepared according to any other than the British Pharmacopœia. There really could be no question on this point, unless it was to be assumed that Acts of Parliament were to be entirely ignored, or taken as of no weight and non-existent. The evidence called for the defendant was simply that of the defendant himself, his manager, the wholesale chemist who supplied him, and the manufacturer. All these, he might say, were in the same boat, and he placed very little reliance on their testimony, for it seemed to him they were in ignorance of the Act of 1862. They might have acted on the knowledge which they previously possessed, but they did not themselves take the trouble to inquire into the present state of the law. He could not himself look at the Act without coming to the conclusion that any drug made up according to any other pharmacopœia than the Britannica was a violation of the Act; and in this view he was confirmed by the decision in the case of "White *v.* Bywater," wherein it was held that if a person asked for tincture of opium he was entitled to be supplied with it of the substance and quality mentioned in the British Pharmacopœia, and not with something of an inferior quality. The magistrate at Sheffield, in that case, at first dismissed the case, holding that in the absence of any special request for the drug according to the Pharmacopœia Britannica, the drug as described in other pharmacopœias could be sold. That was not the view taken by the Judges, who thought the magistrate ought to have convicted the defendant, and who sent the case back to him for re-hearing. He (Mr. Vaughan), therefore, could not entertain any doubt that the article in question must be held to have been not of the nature, substance, and quality of that demanded, and he was bound to convict. But, as he said before, if it could be proved that it was sold as it came from the wholesale manufacturer, he should hold it to be a case in which he could impose a nominal penalty. That had been proved, and he should act on his opinion. Before concluding, however, he hoped that this decision would have the effect of making all persons dealing in drugs remember that the provisions of the Act must be complied with, and that their drugs must be made up according to the British Pharmacopœia. He fined the defendant 40s. and 2s. cost of the summons.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

ABNORMAL BUTTER.

SIR,—Mr. Faber's recollection in connection with myself in the above matter is at fault.

It was distinctly understood that I was to be supplied with more complete details of analyses by Professor Stein, including fatty acids, soluble and insoluble, before I communicated my results to him, at that time a total stranger. I did not consider Professor Stein's Reichert results of any value.

Mr. Allen, though rather ungraciously, withdraws his extraordinary statement *re* Westphal, and I may therefore leave that subject. I may, however, draw attention to the fact that, with regard to the special butter, No. 402, Wigan, *I* was not the authority for the opinion expressed as to its adulteration, an opinion so ably dealt with upon theoretical grounds by Mr. Faber. The analyst for Wigan, and Dr. Jas. Bell, of Somerset House, had already certified the butter adulterated before I received my sample.

I shall be glad if you will find space for these few lines.—Yours, C. ESTCOURT.

Manchester, July 19th, 1889.

BOOKS RECEIVED.

COMMERCIAL ORGANIC ANALYSIS, VOLUME III., PART 1; ACID DERIVATIVES OF PHENOLS; AROMATIC ACIDS, TANNINS, DYES AND COLOURING MATTERS, BY ALFRED H. ALLEN, F.I.C., F.C.S.

THE ANALYST.

AUGUST, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON CONDENSED MILK AND THE ESTIMATION OF CASEIN AND LACTALBUMEN.

BY HARALD FABER.

(*Read at the Meeting, June*, 1889.)

OF the samples of condensed milk I have analysed, which are not many, two may be worthy of the attention of public analysts. One is a sample of a sweetened condensed milk, known generally as the "Goat Brand." It is said to be made from milk, from which "part of the cream" (or fat) has been abstracted. I bought it at 3½d. for a tin containing one pound, which is a lower price than that paid for condensed whole milk, but hardly a low price considering the quantity.

The composition was found to be—

Water	30·91 per cent.
Fat	0·53 "
Total albuminoids	10·83 "
Milk sugar Cane sugar	55·21 "
Ash	2·52 "

From this analysis, I think it may be calculated that the milk from which this condensed milk was made, contained about—

Albuminoids	3·61 per cent.
Fat	0·18 "
Ash	0·84 "

or, that the milk was thoroughly skimmed on a mechanical cream-separator before being condensed, and it appears to me, that the public would better understand the quality of this kind of condensed milk, if it were called "condensed separated milk." An ordinary milk may be taken to contain at least 3·5 per cent. of fat, and it will be seen that there was left barely 5 per cent. of the original fat, or that the "part of the cream" abstracted amounts to over 95 per cent.

The other sample is an unsweetened milk, which, according to the label, is specially prepared for babies. In the directions for use, it is advised to dilute the milk with 7 parts of water for very young babies.

The composition of this milk was found to be—

Water	77·51 per cent.
Fat	6·15 „
Albuminoids	5·35 „
Sugar	9·55 „
Ash	1·44 „

When this milk is diluted with 7 parts of water, the mixture will contain 2·8 per cent. of solids. To recommend such a mixture as the chief or only food for babies might easily have serious, if not fatal consequences.

Unsweetened condensed milk is used, to some extent, in the milk trade, when milk is scarce, in that way, that it is made up with water to its original volume, and no doubt frequently sold as new milk, probably mixed with some new milk. This is done to such an extent that a society of dairy farmers has expressed a wish to put a stop to it, and for that purpose has looked for a method to distinguish between fresh milk and milk which has been condensed and afterwards diluted with water.

Milk contains a genuine albumen, lactalbumen, which, like other albumens, is coagulated when its watery solution is heated to a certain temperature, varying according to circumstances. The more salt the solution contains the higher is the temperature of coagulation, while acids in small amounts lower it. It therefore at once suggests itself that the heating necessary for condensing milk, and especially for sterilising the milk after the closing of the tins, will coagulate the lactalbumen, which is thereby rendered insoluble in water, and that this change in the milk would enable one to distinguish between fresh milk and milk which has been condensed. But, on the other hand, it is generally held, that for some reason or other lactalbumen in the milk is not coagulated when milk is boiled, and, as is a well-known fact, there is no precipitation of coagulated albumen in boiled milk. Some analyses and remarks made a few years back by Professor Hammarsten, to whom, and his pupils, we owe most of our knowledge of the albuminoids in milk, seem to show that the lactalbumen in milk is really altered in boiled, and is at least partly coagulated. As a method was wanted for distinguishing between fresh and condensed milk, I made a few experiments to find whether, and how far, the albumen was coagulated by heating milk, and whether it was possible by analysis to demonstrate such coagulation.

In order to be able to do this, it is necessary to use very exact methods for separating casein and lactalbumen. Concerning the complete precipitation of casein in milk, I think I can do no better than quote from a very exhaustive and exact publication on "The Analytical Estimation of the Albuminoids, with Special Reference to Milk," by John Sebelien, Professor at the Agricultural College at Aas, in Norway, which appeared last year in the transactions of the Royal Academy of Science at Copenhagen:—

"The method of estimating casein in milk first used by Millon and Commaille, and further developed by Hoppe-Seyler, to precipitate it by means of dilute acetic acid in milk diluted with a certain quantity of water, can never give exact results, as the casein is far from completely precipitated by this reagent. The same may be said of sulphuric acid as used by Frenzel and Weyl. Still less exact is the method proposed by Manetti and Musso, to precipitate by rennet.

"A better method is the precipitation by means of sulphate of magnesia, which was perhaps first used by Mitscherlich in 1847, and which, with different modifications, has been adopted by Tolmatscheff, Makris, and Hoppe-Seyler."

Sebelien has made some experiments to ascertain how far sulphate of magnesia, and the equally effective chloride of sodium (which, however, for this purpose must contain some chloride of calcium), give a complete precipitation of casein. He prepared some pure casein according to Hammarsten's method, viz.:—Milk is diluted with about five volumes of water, so much dilute acetic acid is added that the mixture contains a little less than 0·1 per cent. of acetic acid. The casein is precipitated and sinks to the bottom, it is washed by decantation, filtered off, dissolved in as little soda as possible, filtered to get rid of the fat globules, precipitated again by acetic acid, washed, filtered off, again dissolved in very little soda, and so on. The third precipitation is pressed between filter-paper, ground in a mortar with strong alcohol, until it forms a very fine emulsion, then quickly filtered, washed with strong alcohol and afterwards with ether, pressed between filter-paper and ground in a mortar, stirring repeatedly while the ether evaporates. It is finally dried over sulphuric acid, and is then a snowy white very fine powder, which will stand heating to 100° C. in an air bath. It is completely soluble in alkalies, their carbonates and phosphates, in lime—and baryta-water, and even in water in which is stirred up a fine powder of the carbonates of lime, baryta, or magnesia. When the casein is dissolved in a very dilute solution of phosphate of soda, and small quantities of a solution of chloride of calcium is afterwards added, the clear solutions will turn white and milky, looking exactly like skim-milk, and containing the casein in the same state in which it is present in milk, but no trace of other organic matter. Such a solution of casein forms even a skin on top when boiled.

On solutions of pure casein made in this way Sebelien made some experiments. He ascertained the amount of casein in the solution by estimating the nitrogen both in the solution and in the precipitate formed by tannic acid, which he has shown will completely precipitate all the albuminoids of the milk. He then precipitated the casein by sulphate of magnesia and by chloride of sodium—by first adding two volumes of a saturated solution of the salts and then so much of the pulverised crystals of the salts as can be taken up in solution (it is very essential that the solution is completely saturated with the salts). The completeness of the precipitation of the casein was proved, not only by the amount of nitrogen in the precipitate, but by the almost complete absence of any nitrogenous precipitate by tannic acid in the diluted filtrate.

As milk contains a trace of globuline, which is not precipitated by chloride of sodium, but by sulphate of magnesia, and as the precipitates by sulphates of magnesia are more easily treated by the Kjeldahl method, this salt is here to be preferred.

In order to try whether this effective method of precipitating the casein will allow of a correct estimation of the albumen in the filtrate, Sebelien made some experiments on mixtures of solutions of pure casein and pure lactalbumen. Pure lactalbumen may be prepared, according to Sebelien (1885) as follows:—

To the filtrate from the precipitation by sulphate of magnesia of casein in milk (which filtrate is a concentrated solution of sulphate of magnesia), acetic acid is added so as to give ½ per cent. of acetic acid in the solution. The lactalbumen is thereby precipitated; it is filtered off, pressed between filter-paper, stirred up in water (together with the filter-paper), and the water exactly neutralised. The lactalbumen is hereby dissolved. The solution of lactalbumen is filtered off, again saturated with sulphate of magnesia, filtered and precipitated by ¼ per cent. of acetic acid. The precipitate is filtered off, dissolved and dialysed, to remove the salt. The solution thus prepared can be used immediately, or the lactalbumen may be prepared as a powder for future use. In order to do this, the solution is precipitated by alcohol, filtered, washed by alcohol and ether, and ground in alcohol, etc., like the treatment of the casein; it is in this way obtained as a fine white powder, completely soluble in water. It will always retain a trace of the salt, but this will not interfere with the experiments.

Having prepared a milky solution of pure caseine and a solution of pure lactalbumen, and having estimated the nitrogen in both, Sebelien mixed known quantities of the two solutions and analysed the mixture by the following method:—Precipitating the caseine by sulphate of magnesia, washing with a concentrated solution of this salt until the filtrate contained no albumen, then precipitating the albumen in the filtrate after dilution with water, by means of tannic acid or phosphortungstic acid. In these precipitates the nitrogen was estimated (all estimations of nitrogen by Kjeldahl's method) f. inst.

I.—4·180 grms. of a solution of caseine contained 0·01862 grm. N. or 0·445 per cent.
5·742 grms. of a solution of lactalbumen contained 0·01316 grm. N. or 0·229 per cent.

20 c.c. caseine solution was mixed with 30 c.c. lactalbumen solution; this mixed solution contains 0.316 per cent. nitrogen, viz., 0·174 per cent. "caseine N." and 0·138 per cent. "albumen N."

1.—10·125 grms. of the solution gave in the precipitate by Mg SO_4
0·01876 grm. N. = 0·185 caseine N.
In the filtrate, tannic acid precipitated,
0·01428 grm. N. = 0·142 albumen N.
Total .. 0·327 N.

2.—10·180 grms. of the same solution gave in the precipitate by Mg SO_4
0·01750 grm. N. or 0·172 p.c. caseine N.
In the filtrate, tannic acid precipitated,
0·0154 grm. N. or 0·151 p.c. albumen N.
Total .. 0·323 p.c. N.

II.—4·862 grms. of a solution of casein contained 0·01610 grm. N. or 0·331 per cent.
10·193 grms. of a solution of lactalbumen contained 0·00686 grm. N. or 0·067 per cent.

Equal volumes of these solutions were mixed, giving a solution containing 0·200 per cent. N., of which 0·166 per cent. was derived from the caseine and 0·034 per cent. from the lactalbumen.

9·780 grms. of this solution gave a precipitate by Mg. SO_4, containing
0·01568 grm. N. s. 0·160 p.c. caseine N.
In the filtrate, phosphortungstic acid gave a precipitate containing
0·0042 grm. N. or 0·043 p.c. albumen N.
Total .. 0·203 p.c. N.

"The separation of the two albuminoids is as complete as possible, and the amounts found by analysis agree with the amounts calculated as closely as could be expected by an ordinary quantitative analysis."

I have estimated the albumen in some samples of milk by this method, and made some experiments with milk by heating it to different temperatures, in order to find whether the lactalbumen was coagulated, and I found that heating milk to 75° C. will, even in neutral (or amphoter) milk, considerably decrease the amount of lactalbumen found. This must evidently be explained in this way, that the lactalbumen, which is coagulated, although it does not form lumps and sink to the bottom, still is in such a modification that it will be carried down by the casein when this is precipitated, and, therefore, escape estimation as lactalbumen.

The amount of lactalbumen found in fresh milk may vary between 0·35 and 0·45 per cent., and perhaps reach still higher amounts, f. inst., probably in Jersey milk. In boiled milk I found that about the two-thirds of the albumen was coagulated. In the above-mentioned "Goat Brand" sweetened condensed milk, which was slightly sour when opened, and, therefore, most likely was so when filled in the tin, the total albuminoids were 10·83 per cent., of which only 0·085 was soluble albumen, a much lower amount than I have found in boiled and condensed milk which was not acid.

I have analysed an unsweetened condensed milk, known as "The First Swiss Brand," with the following result:—

Water	62·97	per cent.
Fat	10·67	,,
Casein*	9·236	,,
Soluble Albumen	0·324	,,
Sugar..	14·55	(by difference)
Alb.	2·25	per cent.

This milk is evidently condensed to about one-third of its original bulk, which would give these amounts of caseine and soluble albumen in the original milk—

Caseine	3·08	per cent.
Sol. albumen	0·11	,,

I ought to mention that this estimation of albumen was made by boiling the filtrate from the precipitation of the caseine, a method which is not so exact as the precipitations by tannic acid or phosphortungstic acid, as, according to Sebelien's experiments, as much as 12 per cent. of the albumen may remain unprecipitated; still, it is sufficiently exact to show that the milk has been heated, and, therefore, is not "fresh milk."

The low percentage of soluble albumen, if estimated by a sufficiently exact method, will, according to my experience, be a reliable indication, that milk has been heated to at least 75° C., and, as all condensed milk must be heated to at least that temperature to ensure its safe keeping, chemical analysis will be able to prove that such milk is not fresh.

Discussion.

Mr. **Allen** said the paper suggested one point which was a crying scandal, and that was the false and misleading statements made on the labels of some brands of condensed milk. The directions frequently were to dilute the condensed milk with five, six, and even eight times its measure of water, when, as a fact, the milk had only been concentrated to about one-third of its original bulk. The consequence was that if the milk was diluted to the extent directed, and a child were given the amount which would be sufficient in the case of uncondensed milk, it would be half starved. He thought they should not allow these false statements and misleading directions to go unchallenged. Of course, if a case were taken into Court there was the objection that the retailer who sold the can of condensed milk was not the real author of the fraud, but ultimately the offence would come home to the manufacturer, who would find an effect on his trade; and certainly the public ought to be made aware that certain brands of condensed milk are not what they profess to be.

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* Containing the coagulated albumen.

Dr. Vieth said their best thanks were certainly due to Mr. Faber for the paper he had brought before them. They ought to congratulate themselves on the opportunity of reading in The Analyst the very interesting and important experiments of a foreign chemist which Mr. Faber had included in his paper, experiments which originally had been published in a language with which only very few among them were familiar. With regard to the point raised by Mr. Allen, he was of opinion that the total proteids formed the best gauge by which the degree of evaporation could be judged, the proteids being pretty constant and amounting to about 3·5 per cent. As to the mode of distinguishing between fresh milk and milk which had been heated—for this really was the point under discussion—he was of opinion that the determination of the albumen was not a suitable method. Not only was the quantity of albumen present in fresh milk very small, but the separation of the various nitrogenous bodies seemed difficult and uncertain, and the method proposed rather cumbersome. A very simple test for distinguishing between fresh and scalded milk, proposed years ago, consists in adding tincture of guaiacum, which gives a blue colour with fresh, but not with boiled milk. The reaction used to be explained by the presence of ozone in fresh milk, but Babcock, in a recent publication, ascribes it to the presence of fibrine.

Mr. Stokes said that having tried various methods for complete analysis, he found none that constantly summed up to 100 on addition of the constituents. He noticed the same fact in all the complete analyses published hitherto.

In regard to erroneous labelling of tins, he was afraid that no conviction would be obtained. Even in the case of a considerable number of samples prosecuted on his analysis for deficiency of fat, the magistrate held that the samples having come into the possession of the vendors soldered down, so that they could not have tampered with them, no action could lie against the vendors.

The distinction between condensed and fresh milk by means of guaiacum depended upon the milk having been boiled or not. Mr. Stokes, having had charge of, and having practically worked, a vacuum-condenser, knew that often milk was condensed at a temperature not higher than when it left the cow, from 90° to 100° F.; hence the guaiacum test would in such cases fail.

Dr. Voelcker asked Mr. Faber whether he had examined any considerable number of samples of condensed milk, and if so, whether he had found each kind to be uniform in composition. In analysing a large number of one particular brand a few years ago he (Dr. Voelcker) had found great variation in different samples. He understood that for infant life especially, it was important, not only to see that sufficient food constituents were supplied when the milk was diluted, but also that the composition should be always alike.

Mr. Adams said he quite agreed with Mr. Allen and Mr. Hehner as to the importance of not overlooking the constitution of these milks, because there was a point which had escaped remark, and that was the presence or absence of fat, a substance most necessary as a digestive agent. It was by means of the animal fat that true chyle was produced, and the albuminoids were got into the lacteals. He was convinced that a milk hich had a constitution like the first of those given must be a most indigestible ubstance to give any animal, more especially an infant, and that was a reason why the rue facts should be widely known.

With regard to cane sugar in abundance, he considered it, from a medical point of iew, to be injurious. Cane sugar was not easily digested, at all events by young reatures. Of all sources of complaints and disease, a very frequent one was the habit f giving children so many lollipops, and therefore he thought, in the custom of adding ugar, there was injury to the public in being misled as to the extent of dilution these ilks will bear. A child must simply starve if fed on such a milk as the first one, pecially if diluted to seven times its bulk. Animal fat was a most important food—one sense more even than the albuminoids, for without fat the albuminoids could not so well turned to their proper account.

With regard to prosecutions under the Food and Drugs Act, it was a difficult point, o doubt; but he should think there was no doubt that a case of that kind could be ealt with by the law courts.

Mr. Allen asked if a person could be convicted of selling a condensed milk when e had no hand at all in its preparation, and palpably so.

Mr. Adams said it was similar to pickles and green peas, in which cases they had no ifficulty in obtaining convictions. He did not see that that ought to be a great obstacle.

Mr. Stokes said the peas contained an article injurious to health.

Mr. Faber, in reply, said, that with regard to estimating soluble albumen, there as no difficulty when the amount was about 0·3 or 0·4 per cent., and if the amount of luble albumen was so small as to be difficult to estimate, that was by itself a proof that e milk had been heated.

As to the presence of fibrine in milk, he was very doubtful whether that statement ould be found correct after further investigation.

In reference to Mr. Stokes' remarks concerning the temperature of milk during ndensation, it was quite certain you could condense milk at a low temperature, but any case would the milk have to be heated after the closing of the tins to such a egree that the albumen was coagulated in order to sterilize the milk, and thereby sure its keeping for any length of time?

He only knew of very few analyses by this method, as it was comparatively new.

N THE QUANTITY OF VOLATILE ACIDS PRESENT IN BUTTER-FAT.

By Dr. P. Vieth.

(*Read at the Meeting, June,* 1889.)

HE more precise title of my paper would be "On that quantity of volatile acids esent in butter-fat which is determined when butter-fat is examined according to eichert-Wollny's method." The results obtained find expression in the number of bic-centimetres of deci-normal alkali required for neutralising the volatile and, in ater, soluble acids.

When Dr. Wollny, in 1887, published his modification of the Reichert-Meissl ethod for the examination of butter-fat, I in common, I believe, with a great many nalysts, cherished the hope that the method would turn out an extremely useful one. voiding outside influences, which might interfere with the process, seemed to insure rrectness, and working according to fixed and widely adopted rules, promised unirmity of results. The modified method, then, seemed to be well suited for the amination of butter-fat and the detection and quantitative determination of admixed foreign fat, provided the volatile acids present in genuine butter-fat of any origin varied within not too wide limits. According to our experience at the time, we assumed that the volatile acids yielded by 5 grms. pure butter-fat required for neutralisation from 25 to 30 c.c. deci-normal alkali solution. Since then we have learned that those limits were drawn far too narrow, that genuine butters are not unfrequently met with giving results which fall outside, more particularly below, the said limits. The more the matter is investigated, the more cases of undoubtedly genuine butter-fat containing abnormally small amounts of volatile acids are observed.

My own experience with Wollny's modification of Reichert's method commences with a sample of butter-fat sent from Kiel to the members of the "International Committee," formed to investigate into the question of butter-fat analysis. I examined this sample on October 20th (*a*) of last year, and again on January 17th (*b*) and May 2nd (*c*) of the present year, with the following results:—

No. 1. Butter-fat from Kiel (*a*) 31·8, 31·6, 31·8, 31·9, 31·9.
(*b*) 32·1.
(*c*) 32·1.

The insoluble acids in this sample amounted to 87·04 per cent.

The agreement between the results obtained at the various occasions is satisfactory, a fact on which I wish to lay some stress, because not only was, on the several occasions, a fresh soda solution used for saponification and a fresh baryta solution for titration, but also the distilling apparatus employed for the earlier experiments had to be replaced by another one, which was of somewhat different dimensions.

During the time from the middle of November to the middle of June, I applied Wollny's method in the examination of 63 samples of butter as sold in the London market. The results were as follows:—

French Butter.

No.	Date.		No.	Date.		No.	Date.	
2.	14. 11. 88	26·9	12.	23. 1. 89	28·5	22.	27. 3. 89	29·0
3.	23. "	27·2	13.	31. "	27·7	23.	4. 4. 89	29·8
4.	28. "	27·4	14.	6. 2. 89	28·8	24.	9. "	30·5
5.	6. 12. 88	26·8	15.	14. "	29·2	25.	16. "	29·2
6.	13. "	27·6	16.	20. "	29·1	26.	9. 5. 89	30·6
7.	20. "	26·1	17.	22. "	26·4	27.	14. "	30·1
8.	28. "	26·1	18.	26. "	27·5	28.	28. "	29·8
9.	3. 1. 89	26·7	19.	6. 3. 89	29·2	29.	4. 6. 89	30·6
10.	10. "	27·0	20.	13. "	28·7	30.	11. "	29·9
11.	17. "	27·5	21.	21. "	29·5			

Swedish Butter.

No.	Date.		No.	Date.		No.	Date.	
31.	14. 11. 88	27·6	39.	10. 1. 89	27·6	46.	6. 3. 89	27·2
32.	23. "	28·2	40.	17. "	28·6	47.	13. "	28·4
33.	28. "	27·6	41.	23. "	27·4	48.	21. "	28·1
34.	6. 12. 88	29·3	42.	31. "	28·2	49.	9. 4. 89	28·2
35.	13. "	29·0	43.	6. 2. 89	29·4	50.	16. "	28·5
36.	20. "	28·7	44.	14. "	28·2	51.	14. 5. 89	26·9
37.	28. "	27·8	45.	26. "	29·1	52.	22. "	29·3
38.	3. 1. 89	28·2						

Kiel Butter.

No.	Date.		No.	Date.		No.	Date.	
53.	15. 5. 89	29·1	55.	28. 5. 89	27·3	57.	11. 6. 89	27·4
54.	22. "	27·8	56.	4. 6. 89	28·7			

BUTTER MANUFACTURED IN LONDON.

No.	Date.		No.	Date.		No.	Date.	
58.	27. 3. 89	29·2	61.	30. 4. 89	27·6	63.	7. 5. 89	28·7
59.	29. "	29·2	62.	7. 4. 89	28·6	64.	22. "	28·3
60.	30. 4. 89	28·1						

With the exception of the butter manufactured in London, which was churned on premises of the Aylesbury Dairy Company, in Bayswater, little is known as to gin and history of the foregoing samples. The results must all be considered as te normal.

The samples, of which I shall have to speak in what follows, do not refer to butter nufactured for sale. They were, moreover, prepared from small quantities of milk cream in the laboratory under my continuous superintendence, and with exclusion of ry possibility of any admixture of foreign fat taking place. The mode of proceeding s as follows:—One gallon of milk was set in a shallow pan, kept under lock for nty-four hours, the cream taken off and churned, the butter melted, and the fat ered. All the samples form part of a systematic investigation, which was commenced ly in February and continued till the middle of May, and in one direction is still ceeding.

At the end of January a brother analyst asked me for two samples of butter of doubted genuineness. In order to put the purity entirely beyond doubt, I had two aples of cream churned into butter by shaking the cream in glass bottles. Part of butter thus prepared I kept for examination. The one sample referred to cream duced at a butter-factory situated in Sussex; the volatile acids in the butter-fat were nd to be normal. The other sample referred to cream separated on the Aylesbury iry Company's Farm, near Horsham, from milk produced by cows kept on the estate; composition of the butter-fat was to my greatest surprise quite abnormal. I deter- ned at once to try to find out the cause for this abnormal state of affairs, and also to whether anything similar existed with regard to milk produced on other farms at t time.

First of all, another sample of cream from the butter-factory in Sussex was mined. The two samples gave the following results:—

No.		Date.	
65.	Cream from Gl., Sussex ..	2. 2. 89 ..	29·8
66.	" " ..	18. " ..	28·8, 28·3

I next had small quantities of butter prepared from samples of milk received from rty-five farms supplying the Aylesbury Dairy Company at the time. The results e as follows:—

No.		Date.				
67.	P.M. Milk ..	19. 2. 89	from	St.,	Wiltshire ..	27·8
68.	" ..	19. 2. 89	"	He.,	" ..	28·1
69.	" ..	19. 2. 89	"	Fa.,	" ..	30·9
70.	" ..	26. 2. 89	"	Wh. R.,	" ..	30·4
71.	" ..	26. 2. 89	"	Ru.,	" ..	27·9
72.	" ..	26. 2. 89	"	Sm.,	" ..	24·7, 24·8
73.	" ..	27. 2. 89	"	Bo.,	" ..	24·2, 23·9
74.	" ..	27. 2. 89	"	Hw.,	" ..	28·7
75.	" ..	27. 2. 89	"	Da.,	" ..	26·7
76.	" ..	27. 2. 89	"	Ke.,	" ..	25·6

No.		Date.				
77.	P.M. Milk	28. 2. 89	from	Be.,	Wiltshire	29·9
78.	„	28. 2. 89	„	Po. J.,	„	32·4, 31·8
79.	„	1. 3. 89	„	Cr.,	„	26·5
80.	„	1. 3. 89	„	Gr.,	„	26·2
81.	„	1. 3. 89	„	Hi.,	„	25·8
82.	„	3. 3. 89	„	Go.,	„	29 1
83.	„	3. 3. 89	„	Wo.,	„	25·2
84.	„	4. 3. 89	„	Ms.,	„	26·8
85.	„	4. 3. 89	„	Po. R.,	„	28·1
86.	„	4. 3. 89	„	Po. S.,	„	26·6
87.	„	6. 3. 89	„	Mg.,	„	27·2
88.	„	6. 3. 89	„	Kv. Fr.,	„	25·5
89.	„	6. 3. 89	„	Kv. Sh.,	„	31·4
90.	„	19. 2. 89	„	Ph.,	Berkshire	30·8, 30·4
91.	„	26. 2. 89	„	Kv.,	„	28·2
92.	„	28. 2. 89	„	Re.,	„	28·0
93.	„	1. 3. 89	„	Wh. R.,	„	29·5
94.	„	3. 3. 89	„	Je.,	„	27·4
95.	„	4. 3. 89	„	Bu.,	„	27·2
96.	„	3. 3. 89	„	By.,	Cheshire	28·0
97.	„	5. 3. 89	„	Du.,	„	28·2
98.	„	28. 2. 89	„	Ho.,	Oxfordshire	26·9
99.	„	5. 3. 89	„	El.,	„	27·5
100.	„	5. 3. 89	„	Ta.,	Gloucestershire	25·7
101.	„	5. 3. 89	„	Hd.,	Warwickshire	27·2

It will be seen that in two instances the results fell below 25; the lowest and highest figures observed were 23·9 and 32·4 respectively.

I now come to the samples having an abnormal composition. As mentioned before, the milk from which the butter-fat in question was abstracted, was produced by cows kept on the Aylesbury Dairy Company's estate near Horsham. The following were the first results obtained :—

No.		Date.	
102.	Cream from Horsham	2.2.89	20·9, 21·2
103.	„ „	9. „	21·4, 21·2
104.	„ „	15. „	20·4.

The herd which produced the milk consisted at the time of sixty head of cattle, viz., thirty-nine Shorthorn, sixteen Kerry, and five Jersey cows. In order to ascertain whether the breed had anything to do with the abnormal composition of the butter-fat, samples of Shorthorn, Kerry, and Jersey milk were examined separately. The first three samples did not show any striking difference to exist between the three breeds (see the following table), and another point was therefore taken into consideration, viz., the food.

The diet of the cows consisted of suitable quantities of hay, brewers' grains, and linseed—feeding materials which enjoy a very wide application; further, of silage, on average four pounds per head per day; and oatmeal, on average six pounds per head per day. The effect of the two last-named constituents of the diet seemed to me to require elucidation; first, because silage has only since the last few years entered the list of feeding-stuffs for farm live stock; and, second, because the ration of oatmeal is much higher than usually given. After February 18th the Jerseys, and after February

5th the Kerrys ceased to receive silage in their food, while it was always given to the horthorns. The results contained in the table prove that withholding the silage had o marked effect on the composition of the butter-fat. After March 16th silage was iven again to all the cows, but the six pounds of oat-meal were replaced by one pound f barley-meal, two pounds of wheat-meal, and three pounds of linseed cake. From his time the percentage of volatile acids gradually increased, and on the 29th April ould be considered normal in the cases of the three breeds, although approaching the ower limit in two cases :—

No.	Material.	Shorthorns.	Kerry's.	Jerseys.
05, 106, 107	P.M. Milk, 17. and A.M. Milk, 18.2.89	21·4	22·5	20·3
08, 109, 110	,, ,, 24. ,, ,, 25.2.89	22·0	23·9	21·4
11, 112,	,, ,, 3. ,, ,, 4.3.89	22·8	23·1	*
13, 114, 115	,, ,, 10, ,, ,, 11.3.89	21·8	23·1	21·6
16, 117, 118	,, ,, 24. ,, ,, 25.3.89	23·5	24·8	23·3
19, 120, 121	,, ,, 31.3 ,, ,, 1.4.89	24·0	26·0, 26·5	23·6, 24·1
22, 123, 124	,, ,, 28. ,, ,, 29.4.89	25·3	26·1	27·2
25, 126, 127	,, ,, 12. ,, ,, 13.5.89	25·8, 25·9	28·6, 28·6	27·1, 27.2

* Sample lost.

After May 13th no more separate samples of the three breeds were taken, but me sample of cream from the mixed milk of the herd was examined weekly, and I urpose continuing doing so for at least one year. The following results have been btained up to the present :—

No.		Date.	
128.	Cream from Horsham	21.5.89 ..	26·2
129.	,, ,, ...	27.5.89 ..	26·8
130.	,, ,, ...	4.6.89 ..	26·8
131.	,, ,, ...	11.6.89 ..	26·2

One might naturally feel inclined to put the improvement which took place after arch 16th on account of the abandonment of the large ration of oatmeal, and expect further increase in the percentage of volatile acids by giving up feeding cereals altother. To put this to the test, four Shorthorn cows were fed on a diet containing ither oats, nor barley, nor wheat from April 8th to May 2nd; after the latter date ey received six pounds of oatmeal per head per day. The results were as follows :—

	FOOD CONTAINING 1 lb. BARLEY AND 2 lbs. WHEAT.	
No. 132.	P.M. Milk 7. and A.M. Milk 8. 4. 89 ..	26·0, 26·1
	FOOD CONTAINING NO CEREALS.	
No. 133.	P.M. Milk 14. and A.M. Milk 15. 4. 89 ..	24·4, 24·2
,, 134.	,, 22. ,, 23. 4. 89 ..	24·7
,, 135.	,, 28. ,, 29. 4. 89 ..	24·9
	FOOD CONTAINING 6 lbs. OATS.	
No. 136.	P.M. Milk 12. and A.M. Milk 13. 5. 89 ..	26·0, 26·0

The results show that neither barley and wheat nor oats have an unfavourable ect on the composition of butter-fat; on the contrary, in these experiments the latile acids decreased when no cereals were given, and increased after oats had entered into the diet. The variations, however, were hardly large enough to allow any definite conclusions to be drawn from them.

It has been found by several investigators, and also published, that the butter-fat contained in the milk of stale cows, *i.e.*, cows which are approaching the end of the period of lactation, is remarkable for the low percentage of volatile acids which it contains. My own experience on this point is as follows:—

	Shorthorn Cow, Calved 31. 1. 89.	
No. 137.	P.M. Milk 18. and A.M. Milk 19. 2. 89 ..	25·1
	Shorthorn Cow, Calved 16. 5. 88.	
No. 138.	P.M. Milk 18. and A.M. Milk 19. 2. 89 ..	18·6
„ 139.	„ 21. 2. 89 ..	17·6
„ 140.	A.M. Milk 22. 2. 89 ..	17·7
	for ten days' food containing no silage.	
„ 141.	P.M. Milk 13. and A.M. Milk 14. 3. 89 ..	17·9
	Shorthorn Cow, Calved 6. 1. 88.	
No. 142.	P.M. Milk 21. 2. 89 ..	15·0
„ 143.	A.M. Milk 22. 2. 89 ..	16·0
„ 144.	P.M. Milk 1. and A.M. Milk 2. 3. 89 ..	16·3
	for ten days' food containing no silage.	
„ 145.	P.M. Milk 13. and A.M. Milk 14. 3. 89 ..	14·7

The two stale cows gave exceedingly low results, more especially the second one, which, at the time of taking the last sample, had calved sixty-two weeks previously. Sample No. 144 contained 91·28 per cent. insoluble fatty acids.

It remains to be seen whether the low results obtained in February and March cannot be traced to the fact that the cows were then much advanced in the period of lactation. I have calculated the average time elapsed since calving with respect to all the samples, but do not intend troubling you with all the details. The few instances given hereafter will serve as examples, and at the same time prove that, taking into consideration the time elapsed since calving, does not suffice to explain the low percentages of volatile acids found.

No. of Experiment.	Number and Breed of Cows.	Weeks Elapsed since Calving.	Result of Butter-fat Examination.
123	18 Kerrys	18	26·1
120	16 „	27	26·25
106	16 „	26	22·5
120	16 „	27	26·25
122	43 Shorthorns	29	25·3
105	38 „	30	21·4
121	5 Jerseys	40	23·85
107	5 „ .. .	40	20·3

In the examples given, we have in the first and second instance a difference in the times elapsed since calving, but closely agreeing results; in the third and fourth, fifth and sixth, seventh and eighth instances, on the other hand, the times elapsed since calving agree, and the quantities of volatile acids differ.

I have failed, then, to bring home to a certain cause the abnormal composition of the butter-fat produced during the months of February, March, and part of April, and as I do not intend to leave the firm ground of experimental proof, must abstain from entering into speculation on possibilities and probabilities.

Discussion.

Mr. **Faber** said it was a consolation, when so low results were found in butter fat rom cows, to be told that one of them had been in milk for 62 weeks. Of course he ook it that that cow did not give very much milk, and it was not a usual thing in airy-farming to keep a cow so long in milk, and, therefore, he did not think so low results s these from single cows would ever be found in commercial butter. Of course it was f great interest to find how low an amount of volatile acids could be obtained, and Dr. ieth had rendered valuable service by these analyses; but he hoped these results would ot be used to lower still further the figure for calculation which they would have to pply to commercial butter.

Mr. **Allen** asked if Dr. Vieth had made any determinations of the soluble or soluble acids as well as of the volatile acids, because there should be a certain relation etween them, and it would be interesting to know if that occurred in these cases of bnormal butters.

Dr. **Vieth**, in reply, said he had given the result of a very stale cow as being articularly interesting. When results as low as 20 were found in milk from a herd of 0 cows of different breeds, it was a very natural question what would be the limits hen the average was so low. In a herd of cows there must be some individuals giving igh and some giving low results, and with this point in view he analysed these stale nd newly-calved cows. He was rather glad to find this exceptionally stale cow, as it as interesting to see how low the results may fall.

In reply to Mr. Allen, Dr. Vieth said he had made no determinations of the soluble and the total soluble acids, for the simple reason that he had not the time to o it. He made two preliminary experiments with a view to determine the volatile luble and volatile insoluble acids, hoping there might be something in it, but the esults were so discouraging that he gave it up in despair.

He might mention incidentally that the flasks which he used for the saponification nd distillation lost weight at a very uniform rate, the loss on each occasion amounting o from five to eight milligrams.

(Conclusion of the Society's Proceedings.)

NOTE ON A RECENT PAPER BY DR. JOHNSTONE.

By H. Droop Richmond.

t the end of his paper (Analyst XIV. 113), entitled "The Estimation of Soluble d Insoluble Fatty Acids in Butter," Dr. Johnstone gives some equations to represent e action between caustic potash and glycerol, and hints that this action takes place ore readily in the presence of "nascent glycerine" (meaning possibly the hypothetical dicle "glyceryl"). Allen ("Commercial Organic Analysis" vol. ii. 293), has evidently rmed a similar conception to this last; the statement the glycerol on heating with ustic potash is easily converted into a mixture of acetate and formate occurs in the ajority of text-books, and is founded on the observations of Dumas and Stas (*Ann. hem. Pharm.*, 35, 158), who found that by heating solid caustic potash and glycerol gether they could obtain an acid having a mean composition corresponding to that of a ixture of equal molecular proportions of acetic and formic acids, which reduced silver rongly. Redtenbacher (*Ann. Chem. Pharm.* 57, 174), obtained acrylic acid, and Herter und among other products (fermentation) lactic acid (*Ber.* 11, 1167); with regard to ese last, Erlenmeyer's observation (*Ann. Chem. Pharm.* 191, 376), that acrylic acid ves on fusion with caustic potash a mixture of acetate and formate, and Hoppe's

(*Z. f. Physiol. Chem.* 3, 352), that lactic acid is broken up into a mixture of fatty acids, chiefly of the acetic series, shed considerable light on the reaction which takes place, and show that the change is by no means so simple as the equations given assume it to be, but that a series of reactions occur involving first the formation of hydroxy-acids, and their subsequent decomposition, formic, acetic, and other acids being the ultimate products. None of the reactions mentioned above take place at a temperature below 250° C., a fact which negatives the supposition that under the conditions of the Reichert-Wollny process any volatile acid is formed from the glycerol, and that it is not formed from the "nascent glycerine" is shown by the fact that on saponification of olive, earthnut, or other oils, no appreciable quantity of volatile acid can be obtained, even although the oil be heated with caustic alkali for several hours on the water bath, v. Raumer (*Arch. Hyg.*, 1888, 407), however, states that on heating butter for a considerable length of time with caustic potash, he finds an increase in the quantity of volatile fatty acids estimated by Reichert's process, and this is the only fact that tends in any way to justify the supposition that volatile acids are formed during the saponification.

I therefore consider that we have no evidence that the fatty acids distilled over in the Reichert-Wollny process contain any other than those natural to butter, and as, as far as I can ascertain, there is no evidence that "Propionic, Acetic or *Formic* Acids" occur naturally in butter, I should be very glad to know the authority for the statement that they are sometimes present in the distillate.

To turn now to the proposed method. On reading over the paper, I was struck by the seeming fact that the titrations of the insoluble fatty acids after saponification were performed in aqueous solution, and on reference to Dr. Johnstone he very courteously informed me that this was the case; there is no indicator which will give satisfactory results under these conditions, litmus, congo-red, and methyl-orange, showing the whole of the combined alkali, while turmeric paper and phenolphthalein give results varying with the dilution, temperature, point taken as the end reaction and nature of the fatty acids. This is what might have been expected seeing that a neutral soap dissociates in the presence of water, the amount of dissociation varying with the temperature, concentration, amount of other substances in solution, etc. *c. f.* Wright and Thompson (*J. Soc. Chem. Ind.* iv. 630) and Allen (*Co. Org. An.* vol. ii. 245, 260, 265, etc.). The method on this account is totally unreliable.

If from the amount of alkali consumed in saponification (Keottstorfer's figure), we calculate the glyceryl $(C_3H_5)'''$ minus the three atoms of hydrogen already contained in the fatty acids, and add to it the amounts of the soluble and insoluable fatty acids, the total should in butters sum up to nearly 100 per cent. (not quite, owing to the soluble acids not consisting entirely of butyric). The figures given in the paper sum as follows:—

	A.	W.	Y.
Insoluble Fatty Acids	89·95	90·01	89·83
Soluble (as butyric)	7·35	6·26	7·67
Glyceryl—3 atoms Hydrogen C_3H_2	5·18	5·09	5·09
	102·48	101·36	102·69

These are impossible results; that the fault lies chiefly in the estimation of the luble fatty acids is shown by the numbers obtained by substituting the amount of luble acid by the Reichert-Wollny process multiplied by $\frac{100}{90}$ (which gives a very fair proximation to the total amount of soluble fatty acids contained in the butter; this gure is based on the assumption that 90 per cent. of the soluble fatty acids are distilled er in the Reichert-Wollny process, a number which has been shown to be substantially rrect by many observers) *c. f.* Faber (*Anal.* xiv. 98) for the soluble fatty acids; thus:

	A.	W.	Y.
soluble Fatty Acids	89·95	90·01	89·83
-W. × $\frac{100}{90}$	4·86	4·93	5·20
lyceryl—3 atoms Hydrogen C_3H_2	5·18	5·09	5·19
	99·99	100·03	100·22

These figures give a much more satisfactory approach to the correct total, and show at the difference between the soluble fatty acids estimated by the Reichert-Wollny ethod, and those by the method described in the paper are chiefly due to the errors of e latter; should this method be used by Public Analysts upon samples taken under the Sale of Food and Drugs Act," a grievous wrong may be done to the vendor.

The three butters, of which the analyses are given, if genuine, come evidently der the head of abnormal butters. The figures for insoluble fatty acids, viz., 89·95, ·01, and 89·83 respectively, being extremely high, especially with Reichert-Wollny gures of 24·8, 25·2, and 26·6 c.c. (calculated from soluble fatty acids given as by eichert-Wollny process), a fact which points still more to abnormality. If they are nuine, and can be properly authenticated, they should be placed on record as cases of normal butter; it would be most interesting and unusual for three such samples to be the hands of one analyst at the same time.

In his criticisms of the Reichert process, Dr. Johnstone seems to lose sight of the cts that the estimation of the total volatile acids in butter is as arbitrary and as partial figure, as far as the analysis of the butter is concerned, as the numbers obtained by eichert's process, and that Reichert's process is not a method for estimating the volatile tty acids in butter. I maintain that the Reichert method as modified by Wollny and hers is the best and most useful method of butter analysis, on account of its speed and curacy, it being the method by which two analysts working on the same sample will btain the most concordant results; and I contend that the information as to the nature a butter given by this process is at least as accurate and reliable as that given by the timation of the total soluble acids even could these be estimated with absolute accuracy. s to the statement that Reichert's process does not give any insight into the composion of the volatile acids (which, by the way, it does not pretend to do), I should like to now what method of butter analysis does so? A method which gives better informaon than the crude results yielded by Duclaux's fractional distillation process, or the actional liberation of the acids from their salts would be, I am sure, welcomed by nalysts.

ON THE ESTIMATION OF STARCH.

By Prof. Dr. Alex. von Asbóth.

(*Concluded from page* 138.)

Test-Analyses.

No. 1.—*Wheat-flour extracted with ether.*

A. Baryta process.

a. 1·5345 grm.; 10 c c. baryta require 29·75 c.c. acid; 50 c.c. filtrate require 23 61 c.c. = 64·82 per cent of starch.

b. 1·537 grm.; 10 c.c. baryta require 29·52 c.c. acid; 50 c.c. filtrate require 23·42 c.c. = 64 29 per cent. of starch.

c. 1·537 grm.; 10 c.c. baryta require 29·52 c.c. acid; 50 c.c. filtrate require 23·42 c.c. = 64·29 per cent of starch.

Average percentage of starch, 64·46.

B. Lintner's process.

a. 3·032 grm.; 25 c.c. solution reduce ·1995 grm. of copper = 61·99 per cent. of starch. Residue gave blue with iodine.

b. 3·0335 grm.; 25 c.c. solution, reduce ·200 grm. of copper = 62 08 per cent. of starch. Residue colours blue with iodine, and reduces Fehling after treatment with diastase.

No. 2.—*Ground wheat.*

The meal contains 1·78 per cent. of fat and 13·29 per cent. of moisture, and the extracted portion contains 11 per cent. of water.

A. Baryta process.

a. 1·511 grm.; 10 c.c. baryta require 29·52 c.c. acid; 50 c.c. of filtrate require 23·04 c.c. acid = 69·58 per cent of starch = 66·37 per cent. in the original meal.

b. 1·510 grm. 10 c.c. baryta require 29·53 c.c. acid; 50 c.c. filtrate require 23·015 c.c. acid = 69·88 per cent. of starch = 66·65 per cent. in original meal.

c. 2 grm.; 10 c.c. baryta require 29·85 c.c. acid; 50 c.c. filtrate = 21·31 c.c. acid = 70·17 per cent. of starch = 66·97 per cent. in original.

The average amount of starch is therefore 66 65 per cent.

B. Lintner's process.

a. 3·003 grm.; 25 c.c. solution = ·204 grm. of copper = 63·87 per cent. of starch. After treatment with diastase for twelve hours and inversion with 10 c.c. of acid, the residue yielded a further quantity of copper, corresponding with ·83 per cent. of starch.

b. 3·028 grm. Heating was continued for five hours at 145° C. Residue, however, still contained starch. 25 c.c. solution = 202 grm. of copper = 62·74 per cent of starch.

From these analyses we can see that it is impossible to properly estimate the starch by Lintner's process, as the residual mass still contains bodies which may be inverted by diastase.

When I compared the two methods in the analysis of pure starches, and when it was then of course unnecessary for me to use the high pressure flask or to filter the liquids, the results by Lintner's process were certainly satisfactory.

In conclusion, I must draw attention to an article from H. Schreib, who did not succeed in analysing rice by my process, because the rice-starch would not properly gelatinise. The process in its modified form works, however, very well, as will be seen from the following duplicate analysis of a sample of shelled rice :—

Moisture	14·05
Fat	·36
Albumenoids	7·18
Ash	·41
Starch	76·16—75·98
Cellulose	Not determined

ON SOURCES OF ERROR IN DETERMINATION OF NITROGEN BY SODA-LIME, AND MEANS FOR AVOIDING THEM.

By W. O. Atwater.

(*Concluded from page* 96.)

The chief sources of loss appear to be from incomplete ammonification and from dissociation of the ammonia formed.

8. The first of these two difficulties is a serious one. With protein compounds, the great trouble is evidently the formation of volatile decomposition products which do not readily yield their nitrogen to form ammonia with hydrogen. This is easy to understand when we consider that protein compounds are prone to yield cleavage products such as leucine and its congeners, compounds allied to the alkaloids, amido-compounds, etc. ; and that it is very difficult to get all the nitrogen of these latter into the form of ammonia with soda-lime, evidently because of their proneness to form compounds that resist the ammonifying action.

With alkaloids and allied compounds, leucine and other amido-compounds, amines, and azo- and nitro-compounds, complete ammonification is not always effected, even with the utmost care. Concerning peptones our experience does not enable us to speak ; but for the protein of ordinary animal and vegetable substances, including casein, proper precautions appear to insure complete ammonification of the nitrogen. The important condition appears to be sufficient contact with heated soda-lime (*i.e.*, with water vapour at high temperature).

This contact is best secured by (*a*) intimate mixture of substance with soda-lime ; (*b*) close packing, so as to avoid open space inside the tube ; (*c*) providing a reasonably long anterior layer of soda-lime ; (*d*) heating this latter to dull redness before bringing the heat to bear upon the substance, and keeping it hot until the combustion is done. In order to insure the maximum of surface for contact, it is well to have the anterior layer consist of coarse particles of soda-lime containing enough lime to prevent fusing together. It is possible that too long heating may result in expelling the water from the soda-lime, so that there will not be enough in the latter part of the operation to insure complete ammonification.

Though it is desirable to avoid colouration and turbidity of the acid solution, these do not necessarily imply incomplete ammonification, nor does their absence prove perfect combustion. With proper care to insure contact between soda-lime and substance, we have almost never found the solution so coloured as to seriously interfere with titration.

9. Ammonia may be dissociated and nitrogen lost by either too high heating, or by

conducting the operation so slowly as to leave the ammonia exposed for a long time to heat. It seems probable that the presence of water vapour, as of other gases, would tend to prevent dissociation of the ammonia, and that the danger of long heating may be partly due to reduced supply of moisture from the anterior layer of soda-lime after the latter had been heated for some time.

10. A vacant space in the tube (channel as ordinarily recommended) may cause serious loss. This loss is greater the higher the temperature and the longer the time of combustion. It is probably due not only to incomplete ammonification of distillation products through lack of contact with the soda-lime, but also to dissociation of ammonia. With the channel the flow of the gases is slower, and they are exposed to heat longer than when the tube is packed full. Add the possible lack of water vapour when the heating is long-continued and the loss by dissociation is very clearly explained. When the tube is closely packed, the flow of gases reasonably fast, and the operation conducted at a temperature sufficient to heat the tube only to dull redness, there appears to be no considerable loss by dissociation, even with a long anterior layer (20 or 30 cm.) of soda-lime.

Concerning reagents, apparatus and manipulation, a few words will suffice.

1. *Soda-lime.*—The soda-lime made by mixing one part of ordinary caustic soda with two and a half parts of quicklime by the process described, costs very little for materials and labour, and serves the purpose very satisfactorily. In sifting it is conveniently divided into a finer portion to be mixed with the substance, and into coarser particles to be used for the anterior layer. It bears heating without fusing so much as to leave any considerable open space in the tube if closely packed at the outset. Varying proportions of soda-lime, from 1 part to 2½ parts of lime to each part by weight of soda, have made no difference in the results of the analyses. We have obtained equally good results with the mixture of sodium carbonate and slacked lime as described by Johnson, and see no reason why slacked lime, as recommended by him, should not be generally efficacious as it has proven in the cases cited by him and in those tried by ourselves. Our reason for adhering to the ordinary soda-lime has been the impression that by filling the anterior portion of the tube with coarse particles of the rather difficultly fusible material, more complete contact is insured between nitrogenous distillation products and the heated water vapour from the soda-lime. The old theory that enough soda should be mixed with the lime to make the mixture easily fusible does not stand the test of experience.

In testing the purity of soda-lime by sugar, as is sometimes recommended, there is danger of error both from the presence of nitrogen in the sugar and from formation of acid distillation products.

2. *Tubes and charging.*—For ordinary combustions, tubes of from 35 to 40 cm. in length do very well. The method of charging the tube, upon which we have gradually settled after numerous trials with tubes of different lengths and charged in different ways, is explained in the accompanying tabular statement:

Length of tube	40 cm.
Asbestos and fine soda-lime	4 "
Mixture, fine soda-lime and substance	16 "
Rinsings, fine soda-lime	4 "
Anterior layer, coarse soda-lime	12 "
Asbestos, open space, plug	4 "

With a shorter tube the divisions may be made proportionately shorter. We intend to have at least 40 parts by weight of fine soda-lime for every part of substance in the "mixture." About 0·4 and 0·6 of flesh (water free), or corresponding amounts of other aterials, we find convenient. The importance of fine pulverisation of substance, in-mate mixture with soda-lime, filling the tube compactly so as to have no channel, voiding the shaking by which particles of the substance might be brought to the top of he soda-lime, and heating until no charred material is left, has been dwelt upon. An nterior layer of coarse soda-lime, 12 centimetres long, has proven very satisfactory. The nterior layer should be well heated before the heat is applied to the mixture of soda-ime and substance, and kept hot until the combustion is done.

3. *Heat and time of combustion.*—A "low red" heat ordinarily suffices. Heating o bright redness brings danger of loss of ammonia by dissociation, though in our xperience, when the tube is closely packed and the operation not too slow, we have ound practically no difficulty in getting all the nitrogen as ammonia even at high heat. ut with a channel in the tube the loss by high heating has been considerable.

Ordinarily, three quarters of an hour is ample for the combustion, and an hour the xtreme limit, according to the experience in this laboratory. Rapid combustion is less nd long heating more dangerous than is frequently taught. With an ordinary Knop nd Arendt (four bulb) bulb apparatus containing 10 c.c. of acid solution, of which little ver half was required to neutralise the ammonia, the ammonia was completely absorbed ven when the combustion lasted only twelve minutes. Serious loss may result from too ong heating, especially if there be open space in the tube or the temperature is high.

4. *Determination of the ammonia.*—In the combustion of ordinary protein com-ounds with the precautions stated above, practically all the nitrogen is converted into mmonia, and its determination by titration is easy. We find it well, however, to use oncentrated solutions, and to avoid excess of water in rinsing out the bulbs. The uantity of concentrated acid solution required is small, and the tension in the combus-ion tube during the heating consequently slight, which is a convenience; while, ith the small quantity of solution in titrating, the colour reaction is sharp, and the etermination easy and accurate. Freshly prepared cochineal solution is the most satis-actory indicator we have found. Very narrow burettes, in which 10 c.c. occupy form 0 to 40 cm., have decided advantages for convenience and accuracy when concentrated olutions are used.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

"AN EXPERIENCE."

DEAR SIR,—Some time ago you stated your intention of publishing matters of interest in con-ection with prosecutions under the Food and Drugs Act. If the following agrees with your ideas on he subject, perhaps you will be good enough to find room for it.

In May last a sample of whisky was sent to me for analysis from a local borough, and my analysis howed that it was 41° U.P. This result was arrived at, in the first instance, by the Westphal balance, nd then by distillation and determination of the gravity of distillate by a corrected gravity-bottle nd fine balance. The two results agreed within half a per cent. On the strength of my certificate summons was issued against the vendor, whose defence, at the hearing of the case, was that he had ad his portion of the sample analysed by another analyst who made it 18° U.P. In consequence, the hird portion was sent up to Somerset House. The chemists there made it 22·5 U.P. Naturally, the harge was dismissed, and if I had not been very sure of my work, I should have had every oppor-unity of feeling very sorry for myself. Being innocent, however, of any transgression, I made up y mind that on this occasion only the wicked should perish, and I at once set to work to discover ow the business had been managed. I went through my copying books, and found that a sample of hisky had been sent from the same borough in January last. That it was 42° U.P., and that the ndor had paid £1 12s. 6d. for his share in the transaction, and had not appealed to Somerset House. ere was light! and careful search through a capacious cupboard, wherein are kept all empty pack-ges having anything to do with food and drugs, gave me more light, for here I found the January ottle, bearing a label and number corresponding in every way with the label and number on the bottle sent in May, and differing entirely from the labels attached to any other sample sent in May. Two of my staff now went down to interview the Inspector, and they requested a sight of the January sample retained by him. With this, of course, he was unable to oblige them, because, as it was proved further on, he had sent it to me, instead of a portion of the May sample. I now wrote to the Principal of the Laboratory at Somerset House, asking for the bottle he had received to be returned. I was met in the kindest manner possible, and had the satisfaction of knowing that the bottle had been sent to the Justices' Clerk, protected by the official seal. A gentleman from my laboratory now waited upon this same Justices' Clerk to request the privilege of a glance at the label. But to our utter astonishment the seal had been broken, the contents thrown away, and the bottle had been returned to the Inspector ! ! ! I ventured to express my disapproval of this act of the Clerk, and to suggest a different line of conduct upon any similar future occasion. I am sorry to say I gave offence, although I put my protest into the most fatherly language I could command. The Inspector, I am bound to say, gave the bottle up frankly on application, and it was then discovered that I had never even seen any portion of the sample sent to Somerset House, but had re-analysed the January sample. A letter was received subsequently from the Inspector admitting this completely. He said, "I find that when my assistant placed the bottle containing sample of whisky taken on May 4th in the closet, the third portion of sample analysed by you in January, and certified to be deficient in proof spirit to the extent of 17·06 per cent., had been accidentally left in the same closet, and, both being marked No. 1, it is evident that the samples were accidentally transposed."

MORAL.

And so the poor analyst escaped, and his character was not blasted all to pieces; but if he had not looked precious sharp after his own interest, and done his work properly, and kept his sample-bottles tidy, and copied his certificates into a book where he could readily find the same; and if he had not had a decent, honest man to deal with in the person of the Inspector—seeing how largely his show was discounted by the very curious conduct of the Magistrates' Clerk—he would assuredly not have found himself in the satisfactory position of—Yours truly,

PROOF SPIRIT.

To the Editor of the ANALYST.

SIR,—I notice in to-day's *Grocers' Gazette* that Dr. Bernays, Analyst to the St. Saviour's Board of Works, has met with a sample of Condensed Milk similar to the first sample mentioned in my paper on Condensed Milk. It had a label saying that a portion of the cream had been abstracted. The milk was found to contain, when condensed, 0·14 per cent of butter fat, "or about one-twentieth the quantity contained in genuine milk." If the quantity of fat found is not somewhat below that actually contained in the milk, which I think probable, and if the milk was condensed to about one-third, which is likely, as it is recommended to dilute the milk with four parts of water (!), then the "portion of cream abstracted" is no less than 98½ per cent.—Yours truly,

HARALD FABER.

July 13th, 1889.

To the Editor of the ANALYST.

DEAR SIR,—Will you please insert in the next number of the ANALYST the following errata and omissions from my paper on "Fat Extraction":—

P. 122, line 10. After Soxhlet insert (*Ding. Pol. J.* 232, 461).

„ „ 19. After (*Z. f. Anal. Chem.*) insert 1887, 677.

„ „ 6 from bottom, "7, 8, 9, 11, 13 and 15," read "8, 9, and 13," and insert after "methods," 7. The Copper Sulphate method gives less than Adams' (ANALYST, xiv. 88).

11. See ANALYST, x., 46.

15. See ANALYST, iv., 44, and xiv., 89, *Discussion.*

P. 125, line 7, for ·06 read ·04.

„ „ 25, for *Forchngen* read *Forschungen.*

P. 126, line 15, for $\left(\frac{\sigma+\phi}{\sigma}\right)-\frac{G}{D}\frac{1}{\sigma}$

read $T=F\left(\frac{\sigma+\phi}{\sigma}\right)-\frac{G}{D}\frac{1}{\sigma}$

P. 126, line 8 from bottom, after Besana insert (*Ann. Lodi.* 1886, 31).

P. 126, line 18 from bottom, for $T=1{\cdot}17\ F-{\cdot}263\frac{G}{D}$ read $T=1{\cdot}17\ F+{\cdot}263\frac{G}{D}$

P. 127, line 9 from bottom, for *einheittleihr* read "*einheittlichen*"

P. 131, line 7 from bottom, for ·012 read ·01 per cent.

P. 122, line 8 from bottom, for ANALYST xiii. read xii.

P. 125, line 29, for ANALYST viii. read vii.

P. 126, line 6 from bottom, erase from "an examination" to "formulæ," on line 3 from bottom. (This statement was based on calculation with an erroneous formula; on re-calculation I find it is incorrect, and therefore withdraw the statement.)—Yours truly, H. DROOP RICHMOND.

THE ANALYST.

SEPTEMBER, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The annual country meeting was held in Liverpool on Friday, the 19th July. In the course of the afternoon a visit was paid, on the invitation of Dr. Campbell Brown, to the laboratories of University College, after which the members and friends dined together at the Grand Hotel. In the unavoidable absence of the President (who in a letter expressed great disappointment that a pressing demand on his duties as a medical officer prevented his leaving home), the chair was taken by Mr. Allen, among those present being Dr. Campbell Brown, Mr. Norman Tate, Dr. Voelcker, Mr. Smetham, Mr. Thomson, Mr. Embrey, Mr. E. Davies, Mr. Faber, Mr. Duncan, Mr. Bernard Dyer, Mr. Otto Hehner, and several visitors, including ladies. After dinner, in lieu of formal papers, a discussion took place on various professional topics relating more particularly to the work and position of public analysts. On the following morning a visit was paid to the Albert Dock Warehouses and other parts of the Dock Estate, where various specimens of Liverpool imports and exports were inspected, after which most of the party proceeded by boat to Llandudno, remaining at the last-named place until Monday.

(*Conclusion of the Society's Proceedings.*)

A RAPID COLORIMETRIC METHOD OF DETERMINING NITRATES IN POTABLE WATERS.

By Samuel C. Hooker.*

In devising a process for the estimation of nitrates in the minute quantities in which they occur in natural waters, I have not been ignorant of the fact that there are already several very satisfactory ways in which this can be accurately done. I have, however, aimed at greater rapidity than has heretofore been possible.

Fully fifteen years ago, Graebe and Glaser discovered carbazol, and observed that when dissolved in concentrated sulphuric acid, it gave rise to an intensely green solution on the addition of nitric acid. This reaction, which up to the present time has been used only as a means of identifying carbazol, forms the basis of the process to be described. Its extraordinary delicacy renders it particularly suitable for the purposes of water analysis, and enables a quantity of nitric acid, containing as little as $\frac{6}{10000}$ of a milligram. of nitrogen, to be accurately determined. The reaction is not, however, peculiar to nitric acid; all oxidising agents behave similarly. Graebe and Glaser enumerate chlorine, bromine, iodine, nitrous and chromic acids as giving rise to the same intensely coloured solution. With the exception of nitrous acid, the substances just mentioned are almost invariably absent from natural waters, and may therefore be dismissed from further consideration. There is, however, one other oxidising agent, namely iron in the ferric condition, which is sometimes found in very appreciable quantities, and it therefore became necessary, in studying the influence of the substances in solution upon the process, to give special attention to iron as well as to nitrous acid.

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* *American Chemical Journal.*

As the result of a number of careful experiments, the following conclusions were arrived at with regard to the application of the process:

(1) No material error will arise in the presence of nitrites in the quantities in which they usually occur.

(2) Iron (in the ferrous or ferric condition), if present in quantities greater than 0·1 part per 100,000, must be removed.

(3) Chlorides, even when present in very small quantities, must be removed.

(4) Carbonate of lime, sulphates of lime, soda, etc., in the quantities in which they ordinarily occur, were not found to influence the determinations.

(5) The presence of easily destructible organic matter, such as albumen, lowers the result; not materially, however, unless present in large excess.

The estimation, briefly described, is made as follows:—

Two cubic centimeters of the water to be examined is mixed with 4 c.c. concentrated sulphuric acid; the mixture is cooled, and a small quantity of carbazol, dissolved in sulphuric acid, added. If nitric acid is present, the solution will turn green on the addition of the carbazol; and the intensity of the colour, by comparison with that produced by solutions of potassic nitrate of known strength, will, within certain limits, indicate the amount of the acid present.

The great majority of waters can be estimated directly without previous concentration, and only a few will need to be diluted. The limits within which the determinations may be safely made are marked by quantities of nitric acid represented by ·03 and ·40 part N per 100,000. Water containing either less or more nitric acid than indicated by these figures must be concentrated or diluted.

To test the accuracy of the process, a number of solutions were prepared, at my request, containing different quantities of potassic nitrate. In order that my judgment might not be influenced by a knowledge of the amount of nitric acid present, I was kept in ignorance of the strength of the solutions until after the estimations had been completed.

The following are the results obtained:

Nitric Acid expressed as N *in parts per* 100,000.

Present.	Found.	Error.
·127	·130	+·003
·078	·075	—·003
·200	·190	—·010
·302	·300	—·002
·290	·290	·000
Average error - - - - -		·0036

Nitric Acid present in the 2 c.c. of Water operated upon, expressed in Decimals of a Gram.

Present.	Found.	Error.
·00001143	·00001170	·00000027
·00000720	·00000675	·00000027
·00001800	·00001710	·00000090
·00002718	·00002700	·00000018
·00002610	·00002610	·00000000
Average error - - - -		·000000324

The above results are not selected, but include all the determinations that have en made with solutions of unknown strength, with the object of testing the delicacy the process. It is to be borne in mind that potassic nitrate *only* was present. Con- lering the extremely minute quantities operated upon, the accuracy of the above sults is very noteworthy.

The following reagents are required :

(1) Concentrated sulphuric acid.
(2) An acetic acid solution of carbazol.
(3) A sulphuric acid solution of carbazol.
(4) Standard solutions of potassic nitrate.
(5) A solution of aluminic sulphate.
(6) A solution of sulphate of silver.

(1) The sulphuric acid, used for all purposes in the process, should be almost tirely free from oxides of nitrogen. It may be readily tested by dissolving in it a nall quantity of carbazol. If the solution be at first golden yellow or brown, the acid sufficiently pure ; if it be green or greenish, another and better sample must be taken. is essential also that the specific gravity of the acid be *fully* 1·84, and it is ell to ascertain that this is really the case, as I have found several samples, obtained om sources which are generally thought reliable, to fall considerably below these gures. The acid was in fact worthless for the purpose required. As the success of e process depends upon the proper strength of the acid, I cannot lay too much stress pon this point.

(2) The carbazol used was obtained from Kahlbaum, of Berlin. The acetic acid lution is prepared by dissolving 0·6 gram. in about 90 c.c. of 99-100 per cent. acetic id, by the aid of gentle heat. It is allowed to cool, and is then made up to 100 c.c. by e further addition of acetic acid. The exact strength of this solution is of no material portance to the success of the process, and the above proportions have been selected incipally because they are convenient. The solution will probably remain unchanged most indefinitely ; but I cannot speak positively in this respect, as my actual experience tends only over a period of several months. The use of this solution merely facilitates e preparation of that next described, which will not keep, and has consequently to be eshly prepared for each series of determinations.

(3) The sulphuric acid solution of carbazol is easily made in a few seconds, but it is dvisable to allow it to stand from one and one-half to two hours before using. It is repared by rapidly adding 15 c.c. of sulphuric acid to 1 c.c. of the above described acetic cid solution. This quantity usually suffices in my hands for from two to three nitrate stimations. When freshly prepared it is golden yellow or brown ; it changes gradually, owever, and in the course of one and one-half or two hours it becomes olive-green. I m not able to say positively to what this change is due, but it seems probable that races of oxidising agents occur in the sulphuric and acetic acids, and that these, lthough not present in sufficient quantity to act immediately, gradually bring about he reaction described. The greenish colour does not interfere with the process, as might

at first be supposed; on the contrary, I have observed that the solution is not sensitive to small quantities of nitric acid until it has undergone the change to olive-green, and it is for this reason that I have advised that it should be prepared about two hours before required for use. This solution may be thoroughly depended on for six hours after preparation. The intensities of colour produced by the more concentrated solutions of nitrates after this time, gradually approach to each other and become ultimately the same.

(4) The standard solutions of potassic nitrate are very readily prepared. The solutions which are to be compared directly with the waters examined, may be prepared as required, but if many determinations are to be made with a variety of waters, it will be found best to prepare a complete series, differing from each other by ·02 part nitrogen in 100,000. This series may include solutions containing quantities of nitrogen in 100,000 parts, represented by all the odd numbers from ·03 up to ·39. It will be found convenient to prepare them in quantities of 100 c.c. at a time, from a stock solution of potassic nitrate (b) which contains ·00001 gram. nitrogen, or ·000045 nitric acid in 1 c.c. Each cubic centimetre of this solution, when diluted to 100 c.c., represents ·01 nitrogen in 100,000, and consequently if it is desired to make a solution containing ·35 part nitrogen in 100,000 35 c.c. are taken and made up to 100 c.c., and so on. The solution of potassic nitrate (b) is best prepared from a stronger one (a) containing ·0001 gram. nitrogen to the cubic centimetre, or ·7214 gram. potassium nitrate to the litre: 100 c.c. of (a) made up to one litre gives the solution (b). It is obvious that the series of solutions above described could be made directly from (a), but by first making (b), greater accuracy is secured.

(5) For purposes which will be presently described, a solution of aluminic sulphate is required, containing five grams. to the litre. The salt used must be free from chlorine and iron; and the solution should give no reaction when tested, as in the case of a water with carbazol.

(6) The solution of sulphate of silver is required for the removal of chlorine from the water to be examined. It is prepared by dissolving 4·3943 grams. of the salt in pure distilled water and making up to one litre. The sulphate is preferably obtained by dissolving metallic silver in pure sulphuric acid. The solution should be tested with carbazol in the same way as will be presently described for water; if perfectly pure, no reaction will be obtained. As sulphate of silver is often prepared by precipitation from the nitrate, it is very apt to contain nitric acid, and consequently, if the source of the salt be not known, this test should on no account be omitted.

Having described the preparation of the reagents, the method of making the determinations may now be considered more in detail. Assuming the water to be free from all substances capable of affecting the accuracy of the results, the process is carried out as follows: Two cubic centimetres of the water is carefully delivered by means of a 2 c.c. pipette into the bottom of a test-tube; 4 c.c. of sulphuric acid is added, and the solution thoroughly mixed by the help of a glass rod. The test-tube is then immersed in cold water, and when well cooled, 1 c.c. of the sulphuric acid solution of carbazol is added, and the whole again mixed as before. The intensity of the colour is now observed, and a little experience enables a fairly good opinion to be formed of the quantity of nitric acid present. Suppose that the water is roughly estimated to contain about ·15 part nitrogen per 100,000, in such a case, solutions of potassic nitrate containing ·11, ·15, ·19 part nitrogen are selected from the series. Two cubic centimetres are taken from each, and treated side by side with a fresh quantity of the water, precisely as described for the preliminary experiment; the various operations being performed as nearly simultaneously as possible with each of the samples, and under precisely similar conditions. Two or three minutes after the carbazol has been added, the intensity of the colour of each is observed. If at given by the water is matched by any of the standard solutions, the estimation is at end. Similarly, if it falls between two of these, the mean may be taken as representg the nitrogen present in cases in which great accuracy is not required. If this be ne, the maximum error will be ·02 nitrogen, or ·09 part nitric acid per 100,000. If eater exactness be required, or it be found that the colour given by the water is either rker or lighter than that given by all the standard solutions, a new trial must be ade. In such a case the water must be again tested simultaneously with the solutions ith which it is to be compared. This is rendered principally necessary for the reason at the shade of the solutions to which the carbazol has been added is apt to change on anding. Hence it is desirable that the water, and the standard potassic nitrate with hich it is to be compared, should have the carbazol added at as nearly the same time as ssible. When finally the colour falls between that given by any two consecutive members the standard potassic nitrate series, the estimation may be considered at an end, and e mean of these solutions taken as representing the nitrogen present. The maximum ror in this case will be only ·01 nitrogen per 100,000 parts, and the mean error slightly ore than ·005. This degree of accuracy is believed by the author to be quite sufficient r, if indeed it does not exceed the needs of, all ordinary cases of water analysis for sanitary rposes, for which the process was specially devised. If occasionally, however, greater curacy is desirable, the determinations recorded in the early portion of this paper show at the average error can be reduced to ·0036 part nitrogen per 100,000. In this case e standard solutions employed must differ from each other by ·01 part nitrogen only. he additional accuracy gained, however, will scarcely repay for the extra time, care and bour required, and even in this case, as will be seen by a reference to the above deterinations, the error may amount to ·01 part nitrogen per 100,000.

In the course of a great many experiments I have observed, even when the utmost re has been taken, that two solutions, nominally containing the same amount of nitric id, occasionally give a very slight though distinctly perceptible difference in shade hen tested side by side. Whatever may be their cause, such differences are extremely ight, and if proper care be taken, do not materially affect the value of the process.

I cannot too strongly insist upon the necessity of scrupulous cleanliness at all stages the analysis. The quantity of water operated upon is so small, that if the greatest re be not exercised throughout, sources of considerable error may be readily introduced. he test-tubes used in the process may be conveniently $5 \times \frac{5}{8}$ inches in size. They ould, of course, be as nearly the same in diameter as possible. It is scarcely necessary add that they should always be dried when used, and rinsed out carefully with distilled ater when put aside.

In comparing the colour given by a water with that produced by standard potassic trate, it is necessary only to examine the solution *through* the tubes; differences in ade which can only be detected by looking through the length of the columns of liquid, ay be disregarded. It is well to use the same 2 c.c. pipette for measuring both the ater and the standard potassic nitrate; the effect of any error in graduation is thus utralised.

I have made experiments to ascertain whether the intensity of colour produced is rictly proportional to the amount of nitric acid present, with a view to render the use a colorimeter possible, and thus to avoid the time and trouble necessary to match the lours given by the waters. The result of these experiments, which were made with a ubosecq instrument, shows conclusively that the changes already referred to in both tint d intensity after the addition of the carbazol, occur more rapidly with the light than ith the dark solutions, and hence they are not strictly comparable.

Influence of Nitrites.

It has been already pointed out that nitrous acid behaves similarly to nitric its action on a sulphuric acid solution of carbazol. In order to estimate to what its influence would be felt when present in the water to be examined for nitrates, solutions of silver nitrite were carefully prepared and estimated precisely as if th tained a nitrate.

The results obtained are given in the following table:

Results expressed as N *in* 100,000 *Parts.*

Present as $AgNO_2$.	Found.	Error.	Corrected by deductin one-fifth from amount fo
·10	·12	·02	·096
·20	·25	·05	·200
·30	·37	·07	·296

It will be observed that the error may be very nearly corrected by deducti fifth from the amount found. In the presence of nitrates, this correction, of will only be possible in cases in which the quantity of the nitrous acid present h previously ascertained. A correction, however, would be rarely necessary.

Influence of Iron.

Although a solution of ferrous sulphate gives no reaction with carbazol, nitr apt to be overestimated in the presence of iron in the ferrous condition. Ferr however, acting like other oxidising agents, are themselves able to give the charac green colour with carbazol. I have made a number of experiments with solution tassic nitrate in the presence of both Fe'' and Fe''' in varying amounts, and have asce that if iron be not present in quantities greater than 0·1 part per 100,000, the er not exceed ·01 part in 100,000. The condition of a water with regard to iron ascertained sufficiently accurately in a few seconds by the help of ammonic su As this is a test which should always be made in the examination of water for s purposes no extra trouble is involved. In a case in which iron is present i quantities, it can be easily eliminated by rendering the water slightly alkaline, ev ing to dryness, and redissolving the soluble residue in the same volume of pure water as was taken of the water under examination.

Influence of Chlorides.

Chlorides form by far the most serious source of error in this process, by in ing the action of the nitric acid. If, however, nitrates be absent, chlorides give no r with carbazol. Were it not for the fact that the chlorine can be perfectly rem the space of a very few minutes, the general usefulness of the method would b restricted. In order to remove the chlorine I use the standard sulphate of silver referred to, 1 c.c. of which, when consumed by 100 c.c. of water, is equivalent to o of chlorine in 100,000 parts of water. Having first determined the chlorine pre means of a standard nitrate of silver solution of equivalent strength, the exact q of sulphate of silver required is known. I proceed as follows: A flask, graduate neck at 100 c.c. and 110 c.c., is filled up to the 100 c.c. mark with the water to mined. The necessary quantity of sulphate of silver is next added, and then 2 c.c solution of aluminic sulphate previously described; finally, the contents of the fl brought up to the 110 c.c. mark by the addition of pure distilled water. The whole is up and filtered; the first portion of the filtrate, which will contain any foreig ances derived from the filter paper, is then thrown away. The aluminic sulphate, by acting with the carbonates usually present in the water, and thus giving rise to the ecipitation of alumina, facilitates the removal of the precipitated chloride of silver, hich otherwise, under the conditions given above, is extremely difficult to separate, and ay even still pass through the paper after refiltering many times. When the filtrate perfectly bright, a portion is tested to make sure that all the chlorine is removed; e estimation is then made precisely as already described, the amount found being ineased by one-tenth to compensate for the dilution of the 100 c.c. of the water taken to 10 c.c.

It is obvious that such a flask as I have described cannot be used in cases in which e chlorine exceeds eight parts per 100,000, unless the sulphate of silver solution be of eater strength than that which I have recommended. Similar methods will, however, adily suggest themselves.

In a preliminary paper[*] published on this process, I mentioned that probably phenylamine, and other compounds giving similar colour reactions in the presence of ncentrated sulphuric acid, might be used instead of carbazol for the *quantitative* estiation of nitrates in water. Shortly after this paper appeared, L. Spiegel[†] called tention to a process worked up by him, in which diphenylamine was actually used for is purpose. This is briefly described in the *Zeitschrift für Hygiene*, 1887, 2, 189, and as quite unknown to me. Although differing in detail from that which I have devised, e principle is the same, diphenylamine taking the place of carbazol. Spiegel does not ention any experiments made to ascertain the influence of chlorides, but it is probable at this will be found as considerable as I have found it in the case of carbazol.

REICHERT'S BUTTER TEST.

By Dr. Bennett F. Davenport.[‡]

The following minor differences are to be noted between a true butter and a straight oleo, when examined after the Reichert's method.

In the first place, if tasted, the oleo will be found to be deficient in the fulness of flavour of a true butter, while sweet; and it will never acquire the strong flavour and odour of a butter which has turned rancid.

Then when the sample is melted, to allow the curds, water and salt to settle out to the bottom, it will be noticed that the deposit of curds in an oleo will not be more than about one-quarter as much as in a butter. Again, with oleo the saponification is not quite as rapidly completed, and the soap formed is not as rapidly dissolved in the water afterwards added. And when soda is the alkali used, the oleo soap forms a markedly more viscid solution with the water.

Again, in the case of an oleo, a ring of solid deposit forms in the inner tube of the ondenser during the distillation, which never occurs at all with a pure butter.

These differences occurring one after another, will enable the analyst to form a airly reliable opinion as to whether the sample is oleo or butter long before the comletion of the distillation.

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[*] Ber. d. chem. Gessell. 21, 3302; Journal of the Franklin Institute 127, 61.

[†] Ber. d. chem. Gessell. 21, 3568.

[‡] *The Journal of Analytical Chemistry*, Vol. III., Part 2.

THE ESTIMATION OF THE TOTAL ORGANIC NITROGEN IN WATER BY THE KJELDAHL PROCESS.

By Henry Leffmann and William Beam. *

Chemists will note with much satisfaction, through the recent communication of Messrs. Drown and Martin, the successful application of the Kjeldahl method to the determination of the total organic nitrogen existing in waters. The so-called albumenoid ammonia process has never been more than a relative determination, and the estimation by the usual combustion method does not seem to be susceptible of general employment in this field. For the benefit of those unfamiliar with the article in question, we present the following abstract:

500 cc. of the water are poured into a round-bottomed flask of about 900 cc. capacity, and boiled until 200 cc. have been distilled off. The free ammonia which is thus expelled may, if desired, be determined by connecting the flask with a condenser. To the remaining water in the flask, after cooling, is added 10 cc. pure concentrated sulphuric acid. After shaking, the flask is placed in an inclined position on wire gauze, and boiled cautiously in a good drawing hood until the water is driven off and the concentrated sulphuric acid is white or a very pale yellow. The flask is then removed from the flame and a little powdered potassium permanganate added until, on shaking, the liquid acquires a green colour. If the colour is purple instead of green, it shows that the water has not been all driven off. After cooling, 200 cc. of water free from ammonia are added, the neck of the flask being washed free from acid, and then 100 cc. of sodium hydroxide (200 grams. per liter). The flask is connected with a condenser, shaken to mix the contents, and distilled. The 50 cc. are condensed in very dilute hydrochloric acid free from ammonia. The total distillate is made up to 250 cc., mixed, and 50 cc. taken and Nesslerised. A correction for the nitrogen in the chemicals used is made by means of a blank experiment. From the experiments made, Drown and Martin conclude that the nitrates and nitrites do not interfere with the accurate determination of the organic nitrogen, the error which might otherwise be caused disappearing under the conditions of great dilution which exist in natural waters.

Numerous observations have established the fact that the organic matter of water is susceptible of decomposition or volatilisation by simple boiling, often in such a manner that a notable proportion of the nitrogen is lost. It appeared, therefore, in experimenting on this process, that it would be advantageous to add the acid at once, and thus obtain the nitrogen existing as organic matter, and also in ammonium compounds, estimating that existing in the latter in a separate sample by direct Nesslerising, without distillation; the calcium and magnesium which would otherwise interfere with the color reaction being removed by the addition of sodium carbonate and hydroxide, according to the suggestion of Morgan. The same principle can be extended to the determination of the ammonium formed by the sulphuric acid. Instead of distilling the liquid after rendering it alkaline, a little sodium carbonate may be added, allowed to stand, filtered, and the filter Nesslerised. In this way the troublesome distillation is avoided. The procedure is as follows:

Reagents required.—Concentrated sulphuric acid, as free as possible from nitrogen; that which we used was of the same make (Baker and Adamson) recommended by Drown and Martin, and contained a similar proportion of nitrogen (·05 mgm. in 10 cc.).

Sodium hydroxide free from ammonium.—100 grams. of caustic soda are dissolved in about a liter of water, boiled down to about half its bulk in an open vessel, then diluted to one liter with water free from ammonium.

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* *American Chemical Journal.*

Sodium carbonate free from ammonium.—200 grams. pure sodium carbonate are dissolved in a liter of water, and the solution boiled briskly until several hundred cc. have been evaporated.

Determination of nitrogen existing as ammonium.—200 cc. of the water are placed in a stoppered bottle, 2 cc. each of the solutions of sodium carbonate and sodium hydroxide added, the stopper inserted, the solutions mixed, and allowed to stand for an hour or two. A filter is prepared by inserting a rather large plug of cotton in a funnel. This should be washed several times with the ammonium-free water. The clear portion of the sample is drawn off with a pipette and run through the filter, the first portions being rejected, since it is diluted by the water retained in the cotton. The filtration is rapid, and when 100 cc. of the liquid has passed through it is Nesslerised. If the ammonium present is very small in quantity, a rather long and narrow tube should be used for observing the colour.

The following are a few of the results obtained with natural waters, and a comparison with the results by the distillation method.

PARTS PER MILLION.

Modified Process.

	Nitrogen.		
	In NH_4.	Organic.	Total.
Water, already impure, to which decomposing urine was added	0·97	2·34	3·31
Pump-well in densely-populated district	0·24	0·56	0·80
Cellar water—impure	0·17	0·56	0·73

Drown and Martin's Process.

	Nitrogen.			
	In Free Ammonia.	Organic.	Total.	Lost by Boiling.
Water, already impure, to which decomposing urine was added	1·25	1·82	3·07	0·31
Pump-well in densely-populated district	0·33	0·38	0·71	0·09
Cellar water—impure	0·23	0·41	0·64	0·09

In uncontaminated waters much less difference, and sometimes none, was noticed between the results by the original and the modified process.

The figures show conclusively, we think, even if there were no other observations on the point, that it is not safe to estimate the ammonium in the water by distillation, nor to boil the water previous to the estimation of the organic nitrogen.

A SCHEME FOR SEPARATING AND DETECTING SULPHIDES, CYANIDES, FERROCYANIDES, FERRICYANIDES, CHLORIDES, BROMIDES, AND IODIDES IN THE PRESENCE OF EACH OTHER.

BY J. S. C. WELLS AND H. T. VULTE.[*]

Boil out H_2S, test fumes with PbA_2 paper a black coloration indicates H_2S. Add $NaHCO_3$ and distil, carrying distillation to dryness, and pass distillate into KOH, test this for HCy in usual way, when cyanides are removed, cool, dissolve residue in H_2O and add $CuSO_4$ as long as a ppt. is formed, and until the liquid becomes blue or green; filter.

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[*] *School of Mines Quarterly*, X., 3.

Ppt.	Soln.
Cu_2 Fe Cy_6 Cu_3, Fe_2 Cy_{12} NOTE — Test for these acids in the original with Ferric and Ferrous salts.	Add conc. solution of $(NH_4)_2$ Fe_2 $(SO_4)_4$ and boil, pass vapour into starch water. Blue colour indicates HI when this is removed add a drop of conc. H_2SO_4 some dil. $K_2Mn_2O_8$ and boil as long as Br is given off or until the colour is discharged, adding more $K_2Mn_2O_8$ if necessary. Finally test soln. with HNO_3 and $AgNO_3$, a white curdy ppt. sol. in NH_4OH and pptd. by HNO_3 indicates HCl.

NOTE.—So much of the above scheme as relates to separation of Cl Br and I is due to E. Hart (See *Chem. News*, Dec., 1884).

The use of $(NH_4)_2$ Fe_2 $(SO_4)_4$ is original.

ON THE DENSITIES AND REFRACTIVE INDICES OF CERTAIN OILS.

BY J. H. LONG.*

IN the course of a somewhat extended investigation on fats and oils I found it desirable to make full determinations of specific gravity and index of refraction.

For some oils these data are not to be found in the literature, and in most cases where given they are more or less incomplete. In the following pages I present the results obtained with oils of known purity, and will describe first the method of experiment.

Specific Gravity.

This was found by means of a 50 gram. pycnometer with capillary stopper, and was determined at four temperatures obtained by immersion in water in the usual manner. A large volume of water was employed, and its temperature kept constant, as shown by an accurately corrected thermometer. The temperature of the oil in the vessel was known to a tenth of one degree C. The volume and cubic expansion of the pycnometer were determined by special experiments. In the final reductions all weights were calculated to the value *in vacuo*, and the specific gravities referred to water at 4° C. as unity.

The Index of Refraction.

For these determinations I employed the method of minimum deviation, making use of an excellent Meyerstein spectrometer and hollow prism, from which readings to 30'' could be obtained. The refractive index is given by the formula

$$N = \frac{\sin\frac{A+d}{2}}{\sin\frac{A}{2}}$$

in which A is the refractive angle of the prism, and d the observed angle of minimum deviation. Some of the experiments were carried out in a cold room in winter, while others were performed in the summer. To determine the refraction at higher temperatures, the prism, resting on its brass table, was placed on a warm iron plate, and allowed to remain there until a thermometer inserted in the top opening of the prism showed the required elevation. The prism and table were then returned to the instrument, their position was adjusted by means of levelling screws, and the readings commenced. (The arrangement of the apparatus is wn in Wullner's *Lehrbuch der experimental Physick*, II., 129.) By making duplicate eriments, and taking the mean values, results accurate to four decimal places of N obtained. In the following tables the corrected temperature readings are given, the rmometer used being the same as that employed in the determination of density. As rce of light I used the sodium flame obtained by fusing a bead of sodium carbonate r a Bunsen burner.

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* *American Chemical Journal.*

Olive Oil.

We find various statements in the books regarding the density of pure olive oil. a temperature of 15° C. the values vary from ·9120 to ·9250, or even higher. Many mists claim that these limits are too wide for pure oils, but it must be remembered the products are extracted and refined by quite different processes. The hot-pressed contain considerable palmitin, solidify, therefore, sooner, and show a higher density. of a number of samples examined I can report on four of known purity. One of se samples was pressed in Southern California from native olives; the others were ssed in France. Not one of these samples deposited palmitin above 0° C. Although k, they remained perfectly clear, which suggests to me that the statements usually nd in the books regarding the temperature of solidification of olive oil must have been ved from a study of inferior or warm-pressed oils rich in palmitin. Allen, however, s the solidifying point as +4 to — 6° C.

The specific gravity (D) and the refractive index (N) of these oils I found as ows :—

No. 1. Pressed in France.

T.	D.	T.	N.
3·5°	·9240	14·5°	1·4716
19·5	·9131	20·5	1·4693
36·0	·9022	23·5	1·4681
45·9	·8955	26·6	1·4673
		30·7	1·4657
		35·9	1·4637
		41·1	1·4620

No. 5. Pressed in California.

T.	D.	T.	N.
4·5°	·9240	16·3°	1·4712
18·0	·9149	19·3	1·4700
31·1	·9062	23·4	1·4684
49·7	·8938	25·4	1·4677
		29·3	1·4663
		34·3	1·4643
		39·3	1·4622

No. 10. Pressed in France.

T.	D.	T.	N.
2·6°	·9252	15·8°	1·4731
19·5	·9132	20·3	1·4708
33·5	·9039	23·3	1·4694
49·4	·8935	24·8	1·4687
		26·8	1·4679
		30·8	1·4663
		42·2	1·4626

No. 11. Pressed in France.

T.	D.	T.	N.
4·4°	·9238	14·8°	1·4734
19·8	·9128	17·3	1·4722
32·5	·9044	19·3	1·4714
50·6	·8924	21·8	1·4703
		24·3	1·4693
		27·8	1·4677
		30·8	1·4665
		34·3	1·4651

The variation in the specific gravity seems to be the same for all samples, and amounts to about ·00066 for each degree. Schaedler (*Technologie der Fette und Oele n. s. w.*) gives almost the same coefficient for pure samples, and ·9177 as the specific gravity at 15° C. Allen gives ·914 to ·917 as the probable variations. Quincke (Ber. Acad. Ber. 1883, 409) gives 1·4690 as the value of the refractive index at 20·5° C.

This is close to the values found for Nos. 1 and 5, but somewhat lower than those found for Nos. 10 and 11.

The temperature variation of N amounts to nearly ·0004 for each degree between 20° and 35°.

Cotton-seed Oil.

Because of the great practical importance and increasing consumption of this article, I have made numerous tests of samples obtained from different sources. In the last few years great improvements have been made in the processes of refining cotton-seed oil, in consequence of which much of the product now on the market does not correspond to the tests usually given. Cotton-seed oil can be refined (and practically is) until it is as clear and light as water. Such an oil contains few of the colouring or readily oxidisable matters which give the so-called *colour* or *heat* reactions so often employed. The cold test (solidifying point) of such oils is likewise very much altered, and their detection when mixed with other oils made more difficult.

I have obtained the following results by direct experiment:—

No. 7. A crude oil obtained in Chicago from a leading refiner. It deposits a sediment quickly when cooled below 15° C.

T.	D.	T.	N.
3·0°	·9325	27·9°	1·4694
16·6	·9233	35·3	1·4668
32·4	·9126	46·2	1·4624
46·5	·9030		

The specific gravity determination at the lowest temperature is probably somewhat inaccurate.

No. 8. A light yellow oil, refined from No. 7. Remains clear at lowest temperature.

T.	D.	T.	N.
4.5°	·9291	19·3°	1·4744
18·2	·9198	22·8	1·4730
30·9	·9112	24·8	1·4722
45·4	·9014	28·3	1·4708
		36·3	1·4675
		44·2	1·4658

No. 9. A sample refined from No. 8. This is white and clear, and bears no re- lance to ordinary cotton-seed oils.

T.	D.	T.	N.
2·0°	·9312	18·3°	1·4736
17·9	·9202	22·3	1·4720
34·3	·9090	27·3	1·4700
47·0	·9006	31·3	1·4684
		35·3	1·4669
		40·2	1·4650

o. 12. A crude oil, very dark, but deposits a comparatively small sediment on g to 15° C. and below.

The sample is too dark for accurate determinations of the refractive index.

T.	D.	T.	N.
4·8°	·9290	21·0°	1·4708
18·6	·9199	23·0	1·4700
32·5	·9107		
49·6	·8994		

No. 6. A sample refined from the above in the laboratory, by partial saponifica- with a 1 per cent. solution of sodium carbonate, washing, and drying by means current of hot air. The product is yellow, but perfectly clear.

T.	D.	T.	N.
3·8°	·9295	13·8°	1·4755
19·2	·9191	19·3	1·4730
31·4	·9108	23·3	1·4712
47·4	·9000	26·3	1·4703
		32·3	1·4681
		36·2	1·4667
		39·2	1·4660

No. 16. A clear yellow sample, refined in New York.

T.	D.	T.	N.
0·7°	·9322	19·3°	1·4742
15·6	·9219	22·8	1·4728
30·5	·9117	25·3	1·4718
46·5	·9013	27·8	1·4708
		29·8	1·4700
		33·3	1·4686
		36·8	1·4672

The figures given above show that the temperature variations of specific gravity refractive index are not very different from those of olive oil. The decrease in fic gravity at mean temperatures varies in the different samples between ·00066 ·00068, while the variations in the refractive index are something less than ·0004 ach degree.

The values found for the specific gravity of the crude samples are lower than those lly given, as will appear below.

Sesame Oil.

Although this oil is made in large quantities in Europe, it is difficult to secure mens here of guaranteed purity.

No. 4. A sample received from a Chicago importer.

T.	D.	T.	N.
3·3°	·9308	17·9°	1·4744
16·5	·9218	20·4	1·4733
32·4	·9111	24·4	1·4718
48·8	·9001	29·4	1·4698
		32·4	1·4687
		36·4	1·4672
		39·3	1·4661

No. 14. A sample refined from the above by treatment with a 1 per cent. solution of potassium hydrate.

T.	D.	T.	N.
1·5°	·9326	14·3°	1·4771
18·5	·9209	18·3	1·4755
33·2	·9110	22·8	1·4736
47·4	·9016	24·8	1·4728
		26·8	1·4720
		30·3	1·4705
		34·3	1·4691
		39·2	1·4673

No. 18. A sample received from a New York importer.

T.	D.	T.	N.
5·5°	·9282	14·3°	1·4759
18·4	·9192	19·3	1·4739
34·6	·9081	20·3	1·4736
46·6	·8998	23·3	1·4724
		26·8	1·4710
		30·3	1·4694
		34·3	1·4678
		38·0	1·4664

The temperature changes are nearly identical with those given for olive and cotton-seed oil, varying for the density between ·00067 and ·00069 for each degree.

(*To be continued.*)

REGULATIONS OF THE GERMAN GOVERNMENT FOR THE TESTING OF THE MATERIAL USED IN THE DENATURATION OF ALCOHOL.

1. Wood Spirit.

Wood spirit must be colourless or slightly yellowish. On distilling 100 measures under a normal pressure of 760 mm. of mercury, at least 90 measures must come over at a temperature of 75° C. Wood spirit shall be miscible with water without turbidity in all proportions. The amount of acetone shall not exceed 30 per cent. Wood spirit shall decolorise at least 1, but not more than 1·5 per cent. of bromine.

1. Colour.—The colour of the wood spirit shall not be darker than a solution of 2 cc. $\frac{N}{10}$ iodine solution in a litre of distilled water.

2. Boiling-point.—100 cc. of wood spirit is measured into a metal flask; to the flask a distillation-tube furnished with beads is attached, which is connected by a side tube to a Liebig's condenser. Through the upper opening a standard centigrade thermometer is introduced, with the bulb reaching below the side tube. The flask is gently heated so

that the distillate runs from the condenser in drops; the distillate is received in a graduated cylinder, and shall, when the thermometer stands at 75° C. under the normal pressure (760 mm.), amount to at least 90 cc.

If the barometer does not stand at 760 mm., a degree should be added for each 30 mm. For example, at 770 m. 90 cc. should come over at 75·3°; at 750 mm., at 74·7°.

3. Miscibility with water.—20 cc. wood spirit shall give, with 40 cc. water, a clear or very slightly turbid mixture.

4. Separation from soda solution.—On shaking 20 cc. wood spirit with 50 cc. soda solution of 1·3 sp. gr., at least 5 cc. of wood spirit, shall separate in half an hour.

5. Amount of acetone.—1 cc. of a mixture of 10 cc. wood spirit with 90 cc. of water are shaken in a cylinder with 10 cc. of a twice-normal soda solution (80 grs. NaHO in a litre). Next 5 cc. of a twice-normal iodine solution (254 grs. I in a litre) are added with continued shaking. The separated iodoform is taken up by strong agitation with 10 cc. of ether (sp. gr. ·722). 5 cc. of the ether separating after a short time are measured into a tarred watch-glass, by means of a pipette, and allowed to evaporate spontaneously. After standing two hours over sulphuric acid the watch-glass is weighed. The weight shall not be less than ·07 gr.

6. Bromine-absorption.—100 cc. of a solution of potassium, bromide, and bromate (containing 2·447 grs. $KBrO_3$ and 8·719 grs. KBr dried at 100° C in a litre) are mixed with 20 cc. dilute sulphuric acid (one part acid to three parts water). To this mixture, which is a solution containing ·703 grains bromine, wood spirit is dropped in with constant stirring from a burette divided in tenths of a cubic centimetre, till the colour is just discharged. Not more than 30 cc. or less than 20 cc. of wood spirit shall be required.

The bromine-absorption test should be performed by daylight.

2. Pyridine Bases.

Pyridine bases shall be colourless or very slightly yellowish. The amount of water shall not exceed 10 per cent. On distilling 100 measures of the mixture, under a normal pressure of 760 mm. of mercury, at least 90 measures must come over at a temperature of 140° C. The mixture shall be miscible with water in all proportions, without turbidity, and shall be free from ammonia.

1. Colour.—As for wood spirit.

2. Behaviour towards cadmium chloride.—10 cc. of a solution of 1 cc. pyridine bases in 100 cc. of water are mixed with 5 cc. of a five per cent. aqueous solution of fused cadmium chloride, and strongly shaken; a distinct crystalline precipitate must occur almost immediately. With 5 cc. Nessler reagent. 10 cc. of the same solution of the bases should give a white precipitate.

3. Boiling-point.—As for wood spirit, except that at least 90 cc. must have come over when the thermometer reaches 140° C.

4. Miscibility with water.—As for wood spirit.

5. Amount of water.—On shaking 20 cc. of the bases and 20 cc. of soda solution (1·4 sp. gr.) at least 18·5 cc. of the bases must separate on standing.

6. Titration of the bases.—1 cc. of the pyridine bases, dissolved in 10 cc. of water,

are titrated with normal sulphuric acid till a drop of the mixture placed on congo-red paper developes a distinct blue edge which disappears at once.

To prepare the congo-red paper, filter paper is drawn through a solution of 1 gr. congo-red in a litre of water and dried.

[*Note by Abstractor.*—It is in many cases advisable to paint non-bibulous paper on one side with this solution and dry it.]

3. Tar Oil.

1. Colour.—The colour of tar oil must be a black-brown.

2. Boiling-point.—When 100 cc. of tar oils are distilled in the manner directed for wood spirit, not more than 5 cc. shall come over under 90° C, but at least 50 cc. at 180° C.

3. Pyrrol reaction.—2·5 cc. of a one per cent. alcoholic solution of tar oil are diluted with alcohol to 100 cc. On placing a pine-wood chip, wet with concentrated hydrochloric acid in 10 cc. of this solution, containing ·025 per cent. tar oil, a distinct red colour shall develope in a few minutes.

4. Behaviour towards mercuric chloride.—5 cc. of a one per cent. alcoholic solution of tar oil shall, on mixing with 5 cc. of a two per cent. alcoholic solution of mercuric chloride, give immediately a voluminous flocculent precipitate; 5 cc. of a ·025 per cent. alcoholic solution of tar oil mixed with 5 cc. of the mercuric chloride solution shall still yield immediately a distinct turbidity.

4. Turpentine Oil.

1. Specific gravity.—The specific gravity shall be between ·855 and ·865 at 15°.

2. Boiling-point.—When 100 cc. are distilled in the manner directed for wood spirit, not more than 5 cc. shall come over under 150° C, but at least 90 cc. at 160°.

3. Miscibility with water.—20 cc. turpentine oil are strongly shaken with 20 cc. of water, when, after standing, both layers have separated and become clear, the upper one shall amount to at least 19 cc.

5. Ether.

1. Specific gravity.—The specific gravity of ether shall not be more than ·730.

2. Miscibility with water.—20 cc. of ether are strongly shaken with 20 cc. of water. After the separation the ether layer shall amount to at least 18 cc.

6. Shellac Solution.

10 grs. of the solution, on evaporating on the water bath, and drying for half an hour at 100°-105°, shall give at least 3·3 grs. shellac. H. D. R.

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Volumetric Estimation of Zinc. A. Voigt. *Zeitschr f. angew Chemie,* No. 11, 1889.—The sample, which may be any kind of zinc ore or zinc refuse, is dissolved in hydrochloric acid, oxidised with a little nitric acid, and rinsed into a measure with about 100 — 150 c.c. of water. According to the amount of iron present, from 5 to 10 grammes of potassium tartrate are added, the liquid made alkaline with ammonia, and then made up to 250 c.c. Standard solution of potassium ferrocyanide is now added, until a drop of the liquid produces a permanent blue with a drop of glacial acetic acid. The reaction is very delicate and readily noticed. The ferrocyanide solution is prepared by dissolving 46 grammes of the crystals in a litre of water. To standardise this solution, a zinc chloride solution is used, containing 12·461 grammes of zinc oxide per litre. Ten c.c. of this solution are diluted to 150 c.c., mixed with a few drops of ferric-chloride, then with 5 grammes of potassium tartrate, and after being made alkaline, with ammonia, treated as described. If less than 10 c.c. of the ferrocyanide are wanted, this solution must be accordingly diluted, when 1 c.c. will represent ·01 gramme of metallic zinc. This process will give results somewhat too high if the ore contains much manganese, but small quantities of lead do not in the least interfere.

L. DE. K.

VALUATION OF INDIGO. M. HONIG. *Zeitschr f. angew Chemie*, No. 10, 1889.—From ·5 to ·8 gramme of the finely-powdered sample is mixed with about 2·5 grammes of finely-divided dry pumice-stone. The mixture is brought into a Zulkowsky-Wolfbauer extraction apparatus (a kind of Soxhlet), covered first with a layer of powdered pumice-stone, and then with cotton-wool. The extraction is performed with 50 c.c. of anhydrous aniline or nitro-benzol, and is generally completed in an hour. If thought necessary, the mass may be removed from the apparatus, washed with alcohol, dried, powdered, and extracted a second time. The solution is distilled off to recover the aniline, but it is impossible to carry the distillation to dryness on account of bumping. The contents of retort are therefore emptied into a little graduated measure, when the volume must be carefully noted. About five times the volume of absolute alcohol is now added, the precipitated indigotin collected on a weighed filter, washed with alcohol until washings are almost colourless, dried at 110° C., and weighed. To the weight obtained must be added ·013 gramme for every 10 c.c. of aniline left, or, ·021 gramme if nitro-benzol has been used. On the whole, the author prefers using aniline. The indigotin must, of course, be tested for sandy matter. L. DE. K.

TEST FOR LINOLEIC ACID IN COMMERCIAL OLEIN.—K. HAZURA. *Zeitschr f. angew Chemie*, No. 10, 1899.—Fifty grammes of the sample are saponified with alcoholic potash, and after driving off the spirit, dissolved in 1 litre of water, The solution, which must be strongly alkaline, is now slowly mixed with a solution of 50 grammes of potassium permanganate in a litre of water. After about an hour, the liquid is filtered off from the precipitated hydrated manganic dioxide, acidified with dilute sulphuric acid and again filtered. The filtrate is neutralised with caustic potash and evaporated down to about 300 c.c., and then once more acidified with sulphuric acid, which will again cause a precipitate. Without filtering, the fluid is now agitated with ether. If the precipitate dissolves completely, it merely consisted of azelaic acid, and the sample may be considered pure; but if insoluble, linoleic acid is most likely present. To make sure, the precipitate is washed and recrystallised a few times from its alcoholic solution, animal charcoal being added to decolourise. After drying, the melting point is taken, which should come to at least 160° C. If a sufficient amount of this substance should be at disposal, its acidity equivalent may be taken. This should not exceed 150°. By these tests the presence of linoleic acid in commercial oleic acid may be shown with certainty.

L. DE K.

SUBSTITUTE FOR CALCIUM CHLORIDE TUBE IN ELEMENTARY ANALYSIS. JOS. PREUSSER (*Z. f. Anal. Chem.* 28, 322).—Schmitz has described (*Z. f. Anal. Chem.* 23, 515) an apparatus consisting of a U tube containing phosphoric anhydride in the first limb, and sulphuric acid in the second; the P_2O_5 being soon used up, the author 'prefers to place in the first limb a tube containing sulphuric acid, through which the gas bubbles, and to place the phosphoric anhydride in the second. A drawing is given of the tube as made by Gerhardt in Bonn. H. D. R.

The Quantitative Estimation of Thallium and Mercury. W. Feit (*Z. f. Anal. Chem.* 28, 314).—Thallium is generally estimated by precipitation as thallous iodide, but as this salt is not entirely insoluble, the results are not wholly reliable.

Thallic salts react with potassium iodide, forming thallous iodide and free iodine, which the author titrates with arsenious acid solution. The substance is converted into thallic sulphate by the addition of sufficient sulphuric acid, and if necessary the thallous salt oxydised by adding a slight excess of bromine and boiling off the excess; cool, and add an excess of potassium iodide, neutralise the free acid, and add about half as much again of $\frac{N}{10}$ arsenious acid solution (4·942 grs. As_2O_3 in 1 litre) as is required to combine with the iodine, make up to bulk, filter, and titrate an aliquot portion with $\frac{N}{10}$ iodine solution. 506·16 grs. I = 407·2 grs. Tl.

The test analyses are—

Calculated for Tl_2SO_4	Gravimetric.	Voliumetric.
81·12	79·56, 79·47	80·12, 80·14, 80·20, 80·31, 80·31, 80·11

Mercuric oxide is quantitatively reduced by arsenious acid to metallic mercury; the substance to be examined is brought into solution as mercuric salt, precipitated with a slight excess of caustic soda, somewhat more than the necessary amount of $\frac{N}{10}$ arsenious acid solution added, heated, with constant shaking, to boiling, and left to stand for ten minutes, cooled, and made up to bulk, and an aliquot portion titrated after filtration with $\frac{N}{10}$ iodine solution with starch as indicator.

Test analyses :—

Calculated for $HgCl_2$.	Found.
73·80	74·23, 73·73, 74·13, 74·49, 73·89
Calculated for HgO.	Found.
92·59	92·79, 92·45

H. D. R.

LAW NOTES.

Milk of Sulphur and Precipitated Sulphur.

At the Dalston Police Court, before Mr. Haden Corser, magistrate, on August 8th, Mr. Alfred F. Gray, 93 Grosvenor-road, Highbury, pharmaceutical chemist, was summoned by George Morgan, an inspector in the employ of the Vestry of St. Mary, Islington, "for that he did, on the eighth day of July, 1889, sell, to the prejudice of the purchaser, a sample of a certain drug, to wit, milk of sulphur, which was not of the nature, substance, and quality demanded by the purchaser, the said milk of sulphur containing 6·9 per cent. of calcium sulphate, contrary to the provisions of the Sale of Food and Drugs Act 1875, 38 and 39 Vic. c. 63, and the Sale of Food and Drugs Amendment, 1879, 42 and 43 Vic. c. 30, being the statutes in such case made and provided."

Mr. Collingwood, chief sanitary officer, represented the Islington Vestry, and the defendant conducted his own case.

Mr. Collingwood said this was a summons taken out by the Vestry of St. Mary, Islington, against a chemist, and a very respectable one, for selling a sample of sulphur containing calcium sulphate.

Mr. Haden Corser: What does the defendant say ?

Defendant : I plead not guilty.

Mr Collingwood: He must be prepared to fight the analyst, not me. I may say, however, as a preliminary, that Mr. Gray desired that the analyst might be present, and that has been acceded to.

George Morgan was called and deposed that he was an inspector of nuisances for the parish of St. Mary, Islington. On July 8th he went into the shop of defendant, who is a chemist, and asked for a quarter of a pound of milk of sulphur, for which he paid 3d. He told defendant he was an inspector, and divided the sulphur into three parts, giving defendant one portion, keeping one himself, and the same day forwarding the remainder to the public analyst.

tificate of the public analyst was here put in. The analyst stated that the sample of milk eceived by him weighed about 1½ or., and contained 6·9 per cent. of calcium sulphate, to e of the purchaser.

lingwood : I may tell your worship that this milk of sulphur is a powder.

len Corser : Thank you ; I already knew it. (Laughter.)

lingwood : This calcium sulphate our analyst alleges to be an adulterant, and he is present is certificate.

, in cross-examination by Mr. Gray, said he asked for milk of sulphur. He did not ask for sulphur, because he did not want it.

nt : Did I look at this book (produced) to see what to charge you ?—Yes.

nt (to the magistrate) : In this printed book it says, "milk of sulphur, 3d.," and "pre- phur, 4d." the quarter-pound. This was distinctly sold as milk of sulphur, and not as (To the inspector) : Do you know the difference between the two substances ?.—No.

lingwood : He would not know. He was merely instructed to buy milk of sulphur. If his ler the circumstances, sees a way for you to get out of your difficulty we have no objection.

nk Teed, 8 Victoria Chambers, Westminster, put in his certificate of appointment as st for the parish of St. Mary, Islington. He said : I have examined the milk of sulphur fendant's shop, and find it to contain 6·9 per cent. of calcium sulphate. Milk of sulphur ate of sulphur are one and the same thing, or least they ought to be.

nt : What authority have you for that.

gistrate : One moment. Can you ask for the one or the other ?

lingwood : In asking for milk of sulphur the inspector was right, as proved by the fact it.

d : Any extraneous matter in the sulphur is an adulterant. I say the milk of sulphur and ted sulphur are one and the same thing. I take the extract from Watt's "Dictionary of

gistrate : I cannot take that. But you know what these substances are ? You say that sulphur should contain nothing but sulphur. I see there are different prices for the two list.

nt : I suppose you know, Dr. Teed, that milk of sulphur is in the old Pharmacopœia, if it new ?

d : It is not in the present one.

nt (producing old book) : It is in this, the old Pharmacopœia ; and ever since it was laid book the two articles have been manufactured and sold as distinct articles.

gistrate : You say that you sell them in different ways and as distinct articles ?

nt : Yes ; there is lac sulphuris and precipitated sulphur. (To the analyst) : Are these stinct articles ?

d : I should think not.

nt : This case has been brought up before, your worship, so far back as 1877, at Knutsford, oratio Lloyd, chairman of the bench of magistrates, decided the matter in the following Ve have been considering the matter since we left the court, and——"

gistrate : You are not quoting from a law report ?

nt : No.

gistrate : You may read that, and ask the witness if he agrees with it.

nt (reading) : "The case having been fully gone into, the Chairman said, 'We have been this matter since we left the court—and I am speaking for all the magistrates who heard e think the absence from the Pharmacopœia of milk of sulphur justified the complainers ese proceedings ; but we find that milk of sulphur and precipitated sulphur are supplied to two distinct things. We cannot doubt upon the evidence that the offence has not been I submit, therefore, there is no case. Will your worship allow me to ask the analyst if pt the verdict of the Pharmaceutical Society on the matter ?

gistrate : No. Give him the opinion, and ask him if he agrees with it.

nt (to the analyst) : Do you accept what the old Pharmacopœia lays down in the matter ?

d : The old Pharmacopœia is not allowed to be used now, and the two things are not in the copœia.

nt : But it does not follow that the two things are the same.

to the Magistrate, Dr. Teed said : For some years the two things have been known under

Collingwood : I quite understand that the milk of sulphur and the precipitated sulphur the same thing. The title has been dropped in the one. Milk of sulphur is the chemical cipitate of sulphur.

gistrate : The defendant can go into the box and give evidence.

y was therefore sworn, and said : The article I sold is milk of sulphur. I was asked for supplied the article which answers to the description.

gistrate : Did you know the composition of the article you were asked for ?

nt : It is a compound containing calcium sulphate. In the process of manufacture it is o remove the whole of the calcium sulphate.

The Magistrate: Is the milk of sulphur manufactured by yourself?

Defendant: No. I purchased it.

The Magistrate: Was the purchase in accordance with the rules of the Pharmacopœia?

Defendant: Yes, under the old one. Lac sulphuris is the old one which I have here. The new Pharmacopœia has only precipitated sulphur. I sell the articles at different prices, and have them supplied the same.

Mr. Collingwood: Is it not a fact that the old Pharmacopœia has been declared illegal?

Defendant: Not to my knowledge.

Mr. Collingwood: I am told that in a recent case affecting sal volatile the old Pharmacopœia was declared to be illegal.

Defendant: I can explain that. Sal volatile in the new Pharmacopœia is ordered to be made in a different manner to the old. That is the standard work which is accepted as an authority.

The Magistrate (to Mr. Collingwood): What I understand you to say is this, that because milk of sulphur is not in the new Pharmacopœia it should not be supplied at all?

Mr. Collingwood: Precipitated sulphur should be given. (To Defendant): I think you said in your evidence that it is impossible to separate the calcium sulphate from the precipitated sulphur, and then you say you sell both articles.

The Magistrate: He says in his judgment they differ.

Defendant: It is precipitated by hydrochloric acid.

Mr. Collingwood: Cannot the sulphate of calcium be washed out?

Defendant: That is not the way in the new Pharmacopœia.

By the Magistrate: I cannot say if it can be all washed out. It might in time all get washed out.

The Magistrate: What is the object of keeping the two sorts?

Defendant: Because people will have what they ask for. And plenty of doctors will give evidence that milk of sulphur is more soluble and more beneficial.

Mr. Collingwood: It is not known in the new Pharmacopœia. That is our case.

The Magistrate: Is the British Pharmacopœia absolutely binding upon chemists?

Mr. Collingwood: I believe it is, sir.

The Magistrate: If this gentleman had been asked for an article which is not in the British Pharmacopœia, but which according to his judgment he supplies, is there anything to prevent him? —No reply.

The Magistrate: If he was asked for milk of sulphur, should he say he had not got it?

Dr. Teed: He should have supplied the precipitated sulphur.

The Magistrate: I should like very much the judgment of a superior court on such a matter as this. This vestry official goes in to purchase an article which he knows he will not get according to his own views. You (addressing Mr. Collingwood) knew defendant sold the two kinds probably. He asks for and gets what defendant calls milk of sulphur, and Dr. Teed says that is not known in the British Pharmacopœia. But the defendant says, and Dr. Teed does not deny, that it is in the old Pharmacopœia, which you say is now obsolete.

Mr. Collingwood: I will not further trespass on the time of the court, but leave the case in your worship's hands.

The Magistrate: And I need not enter into a lengthy judgment in the case. Defendant has been asked for an article known as milk of sulphur, and the purchaser gets what manufacturers and the public know as milk of sulphur——

Mr. Collingwood: I am afraid the public do not know the composition of it.

The Magistrate: I hold that he has not got this article to the prejudice of the public, and shall dismiss the summons. He had the precipitate of sulphur as well if anybody wanted it, and the prices of the two articles were different.

Mr. Collingwood: We have had milk of sulphur over and over again which has not contained calcium sulphate.

Defendant asked for his costs, but the magistrate thought there was no necessity for allowing them.—*Chemist and Druggist.*

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

Note (?) in a recent paper by Dr. Johnstone.

SIR,—I have neither the time or inclination to commence a controversy in the pages of the ANALYST with such a young and inexperienced hand as Mr. Richmond, more than to state that the three samples of butter were, without doubt, genuine, and through your kindness they have already been "recorded" in the ANALYST. I also most emphatically deny ever having informed your self-opinionative contributor, who is strong in the infallibility of his own judgment, that I titrated the insoluble acid in an aqueous solution, and I can assure him that dissociation did not take place in the soap solutions titrated by me.

Yours faithfully,
WILLIAM JOHNSTONE.

Crieff,
23rd August, 1889.

THE ANALYST.

OCTOBER, 1889.

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A NEW VOLUMETRIC METHOD FOR THE ESTIMATION OF FAT IN MILK, SKIMMED MILK, BUTTER-MILK, AND CREAM.

By Charles L. Parsons.

There have been, within the last few years, so many improvements in breeds of cattle and in the production of milk, butter, and cheese, that no creamery-man, dairyman, or farmer can ignore them and still hold his own against the increasing competition of the day. Breeds of cattle and individual cows must be kept which pay for the food they consume by their milk, or they must be turned into beef. Butter must be made which comes up to the standard in flavour, colour, and grain. Milk must be sold which meets the requirements of the law, where it is to be consumed as such, and where it is to be used in making butter, quality as well as quantity will soon determine the value. But, one may ask, on what basis shall one cow be kept and another rejected, or milk be bought and sold? The question has been many times answered. Quantity was formerly the basis, but now the amount of butter-fat which the milk contains must have equal consideration. The quantity is easily determined by measures or scales, but to determine the fat has been quite another matter.

Every butter-maker, in order to compete with others, must soon know the amount of fat in the original milk, the amount he has obtained in his cream, and the amount lost in the skimmed milk and buttermilk. There has long been an increasing demand for some quick, simple, cheap, and accurate method which is applicable to all four.

Butter-makers have been satisfied with general deductions from analyses at some distant laboratory, but now, from the demonstrated differences of the effect of individuals, breed, food, and temperature upon the churnability of cream, they must be made often, and at home.

Different herds vary greatly in the quality of milk which they produce. This is shown plainly by the results of analyses given in the second annual report of the New York Dairy Commission. The average milk of seventy dairies there reported varied between 3·08 per cent. and 6·60 per cent. of butter-fat. It is manifestly unfair to every creamery and progressive farmer that the same price should be paid for milk containing three per cent. of fat as for milk containing double that quantity.

Many methods have been devised to be used for "testing milk." Feser's lactoscope, the lactobutyrometer, the creamometer, the oil-test churn, Heeren's milk-tester, the methods of Vogel, Soxhlet, and Short, and the lactocrite have all been proposed for general use and have met with varied success. Of these, the methods of Soxhlet and Short, and the lactocrite, are undoubtedly the best. The expense of the lactocrite, and the ether required by the method of Soxhlet, however, shut them out from many creameries and dairies. Short's method requires no great expense, but while it gives good results on whole milk, in the words of its author, it does not give "accurate results with milk containing under ·50 per cent. of fat." This, of course, shuts out the analysis of most skimmed milk and some buttermilks. If a butter-maker is to balance accounts, the fat lost in the skimmed milk and buttermilk must be taken into consideration, as well as the fat in the milk and cream.

I have for some time past been experimenting for the purpose of obtaining a simple and cheap method, applicable to all grades of milk and cream, which would give good results in the hands of any dairyman. The following is the method obtained, and no scientific terms have been used where they could possibly be avoided:—

THE APPARATUS.

1. (*a*) Three pipettes. No. 1, to hold 100 cubic centimeters; No. 2, to hold 50 cubic centimeters; No. 3, to hold 25 cubic centimeters, and having the lower stem at least nine inches long. (*b*) A measuring-cylinder holding, when full, 10 cubic centimeters. (*c*) A measuring-tube nine inches long and of such an internal diameter that the distance on the scale from zero to five shall be between seven and one-half and eight inches. These tubes are accurately calibrated, and are marked on the outside. The numbers 1, 2, 3, 4, and 5 on the scale represent so many whole cubic centimeters. The distance between 0 and 1, 1 and 2, etc., is divided by twenty lines, the distance between two of these subdivisions representing ·05 of a cubic centimeter.

2. One or more long, slender bottles made of transparent glass, eleven inches high and one and one-half inches in diameter, which hold about 250 cubic centimeters. The neck should fit a No. 2 rubber cork.

3. One or more flasks of about 70 cubic centimeters' capacity, with the neck cut off obliquely, and of a size and length to go into the top of the measuring-tube.

4. A drying-oven, eight inches wide, twelve deep, and fourteen high, which has a

thermometer, shelf for holding flasks and measuring-tubes, and a single-burner kerosene stove beneath for heating.

SOLUTIONS REQUIRED.

1. Gasoline, practically free from anything in solution. This gasoline is that ordinarily used in gas machines. It is sold in three grades (86°, 88°, and 90°), either of which will do. It begins to boil at about 80° F. and generally the last portions boil off at 140° F. (The substance used in street lamps generally called gasoline is not such, but naphtha, and will not do.)

2. Caustic soda solution. This is made by dissolving in an iron vessel one pound of commercial caustic soda in two pounds of water.

3. Strong alcohol (95 per cent.), in which one ounce of Castile soap to the gallon has been dissolved. To dissolve easily the soap should be scraped fine.

4. A little strong acetic acid.

DIRECTIONS FOR ANALYSIS.

Fill the largest pipette to the mark with the well-mixed milk, and run into one of the bottles. Add to it the measuring-cylinder full of caustic soda solution and half full of alcohol soap solution. Then add pipette No. 2 full to the mark of gasoline. Cork the bottle. Shake hard for a few seconds, and again five or six times, at about equal intervals, in the next half hour. After the last shaking let the bottles stand a few minutes to see if the gasoline will not rise to the top. If it does not, add the measuring-cylinder half full of alcohol solution again, and as soon as bubbles cease to rise, turn the bottles slowly upside-down and back again. This may be repeated until four portions of alcohol have been added, or separation takes place sufficiently to draw out the required quantity of the upper liquid. The bottles should be turned slowly in order not to mix again any of the gasoline solution which has separated. This separation generally takes place after the first addition of the alcohol solution.

As soon as the upper solution is perfectly clear, fill the smallest pipette to the mark with it, taking great care that none of the lower solution enters. From the pipette let the solution run into one of the flasks and evaporate the gasoline. Add two drops of acetic acid to the fat which is left behind. Place the flask in the oven and dry it at 245° to 255° F. for one hour and a-half. Now turn the fat slowly into one of the measuring-tubes previously placed in the oven. Let the flask drip for ten minutes while the oven is at the same temperature. Remove the last drop by drawing the neck of the flask across the neck of the tube. Cool the measuring-tube until the first appearance of the fat solidifying, hold clasped in the hand a moment, and then read off the number on the scale which corresponds with the upper surface of the fat. From the accompanying table ascertain the per cent. of fat which corresponds to this number.

Several analyses can be carried on at once, all flasks, bottles, and tubes being carefully numbered.

SKIMMED MILK AND BUTTERMILK.

Skimmed milk and buttermilk are treated nearly the same as whole milk. The fat from either, if less than 1 per cent., need be dried only three quarters of an hour. If more than 1 per cent. it should be treated as a whole milk. It is easy after analysing one or two milks to judge approximately from the amount of dried fat in the small flasks whether it is above or below this per cent. If the milk is thought to be below 1 per cent., it should be measured in a tube which is already at least half full of dried fat. The reading on the scale for the fat already in the tube should be taken, and the reading after the addition of the fat from the flask. The difference between the two gives, of course, the reading for the skimmed milk or buttermilk.

CREAM.

The sample should be carefully mixed, and free from air bubbles. Fill the largest pipette to the mark and let it run out into some suitable vessel. After blowing out the last drops, fill the same pipette with water and mix this well with the cream already measured. With this mixture fill pipette No. 2 to the mark, run it into one of the bottles, and proceed as for whole milk. The fat from very rich cream should be dried for two hours. It is always best to carry on two analyses of milk or cream, whatever method is used, the one to check the other. Results cannot be expected to agree as closely on cream as on milk, for the amount used is only one-fourth as great; nor is the accuracy, necessary in the one case, required for practical work in the other.

DETAILS.

Sampling.—The milk or cream should be well mixed and free from air bubbles. Fill the pipette a little above the mark on the neck by suction with the mouth, and quickly place a finger (dry) over the top. Then, by gradually letting in air, allow the milk to run out slowly until it just reaches the mark. All solutions measured with a pipette should be measured in this manner. These measurements should be made accurately, especially the measurement of the gasoline solutions. Always allow the pipettes to drip a moment, and blow out the last drops. In analysing milk in creameries where the milk is tested only at intervals, the samples should be as nearly as possible an average one. To this end, mix the night's and morning's milk.

Treatment of the Gasoline Solution.—Do not try to hurry the separation, as too much alcohol soap solution is worse than too little. The separation of whole milk or cream generally takes place within ten minutes. Skimmed milk and buttermilk occasionally require a longer time, but will always separate. Sour buttermilk does not separate as easily as fresh, and sometimes has to stand over night. If in filling pipette No. 3 any of the lower solution enters, the whole must be run back into the bottle and allowed to settle again. This need never occur if care is taken to draw up the solution slowly and not by jerks. Before using the small flasks for the first time, each one should have a little pure fat put in and melted. This should be allowed to drip out under the same conditions as in actual analysis. (Butter which has been melted and the salt and curd allowed to settle out, or the fat obtained in analysis, is best for this purpose.) A film of fat is thus left behind, which compensates for the fat left behind in analysis. This film is always approximately the same, as in evaporating the gasoline it is each time washed down by the gasoline condensing on the sides of the flask, and is again left when the fat is turned into the measuring-tube.

Evaporating Gasoline.—Gasoline takes fire very easily, and should not be evaporated within several feet of a flame, unless protected. It boils much below the boiling point of water. The best way for general use is to heat a pan or pail of water to boiling, and then carry it some distance from the fire. Now take the little flask con-ining the gasoline and fat in the hand and hold it on the hot water. The gasoline ill immediately begin to boil, and in a short time will mostly pass off into the air, aving the fat and a little gasoline behind. Care must be taken that the gasoline does ot boil over, for in that case some of the fat would be lost. If it shows any tendency do this, lift the flask from the hot water a moment, and the boiling will stop. The ater should not be allowed to cool too much before evaporating. The boiling gasoline ill not burn the hand, as it boils at too low a temperature. The acetic acid is added prevent the fat becoming gelatinous, as it frequently does without it. In creameries here steam is always to be had, a large number may be evaporated at once. Bore some les in the top of a small closed box; these should be about the size of the ttom of the flasks. Place the flasks containing the gasoline and fat on top of ese holes, and pass steam very slowly into the box through a rubber pipe. This is by r the easiest way, and requires only about five minutes for any number desired. The ow of steam must be under control, so that it may be stopped at any moment should e gasoline boil so fast as to be in danger of boiling over. Only a very little steam required.

Drying the Fat.—The flasks containing the fat are placed on the shelf in the oven. he thermometer bulb should be on a level with the flasks. The oven is kept between 5 and 255 degrees Fahrenheit, and the flasks are dried at this temperature for the ll time. If for more than five minutes the temperature should fall below 245, the sks should be allowed to stay enough longer to make up for time thus lost. The sks should drip into the measuring-tubes full ten minutes, with the temperature at e required height.

Reading the Measuring-Tubes.—After the fat is ready to measure, the tube is cooled wn in the air, or, by taking care that none enters the tube, with water. As soon the fat begins to grow opaque through solidification, the part of the tube containing t is held clasped in the hand a moment until it clears up, when the reading is taken, hich corresponds with the upper slightly curved surface of the fat. Considering 1 on e scale as 100, 2 as 200, 3 as 300, etc., each sub-division equals five. This is perhaps e best way to read the scale, as decimals are avoided.

Cleaning the Apparatus.—Always rinse out pipette No. 1 with water, and pipette o. 3 with gasoline, when through using them. To clean pipettes, thoroughly fill them ith some of the caustic soda solution, taking care that none of it gets in the mouth, d let them stand some time. Then turn it back and rinse well with water. Pipettes os. 2 and 3 should be dried before again using for gasoline. The flasks should be eaned in the same way whenever necessary. It must be remembered, however, that film of fat is intended to remain on the inside, and as long as nothing but fat is esent, cleaning is unnecessary. The tubes are cleaned carefully after each analysis by arming them, turning out the fat, and rinsing each one twice with gasoline while still arm. Dry before using.

Using the Tables.—Find the first two figures of the reading obtained from the scale the left hand column headed c. c. Then pass horizontally towards the right to the lumn which has the third figure of the reading for its heading, and the per cent. of t corresponding to the reading of the scale is found.

TABLE

FOR MILK, SKIMMED MILK, AND BUTTERMILK, CONTAINING FAT FROM ·18 PER CENT. TO 8·56 PER CENT.

c.c.	0	1	2	3	4	5	6	7	8	9
0·1	·18	·20	·22	·24	·26	·27	·29	·31	·33	·35
0·2	·36	·38	·40	·42	·	·45	·47	·49	·51	·53
0·3	·54	·56	·58	·60	·62	·63	·65	·67	·69	·71
0·4	·72	·74	·76	·78	·80	·81	·83	·85	·87	·89
0·5	·90	·92	·94	·96	·98	1·00	1·02	1·04	1·06	1·08
0·6	1·09	1·11	1·13	1·15	1·17	1·18	1·20	1·22	1·24	1·26
0·7	1·28	1·30	1·32	1·34	1·36	1·37	1·39	1·41	1·43	1·45
0·8	1·46	1·48	1·50	1·52	1·54	1·56	1·58	1·60	1 62	1·64
0·9	1·65	1·67	1·69	1·71	1·73	1·75	1·77	1·79	1·81	1·83
1·0	1·85	1·87	1·89	1·91	1·93	1·94	1·96	1·98	2·00	2·02
1·1	2·04	2·06	2·08	2·10	2·12	2·13	2·15	2·17	2·19	2·21
1·2	2·23	2·25	2·27	2·29	2·31	2·33	2·35	2·37	2·39	2·41
1·3	2·43	2·45	2·47	2·49	2·51	2·53	2·55	2·57	2·59	2·61
1·4	2·63	2·65	2·67	2·69	2·71	2·73	2·75	2·77	2·79	2·81
1·5	2·83	2·85	2·87	2·89	2·91	2·93	2·95	2·97	2·99	3·01
1·6	3·03	3·05	3·07	3·09	3·11	3·13	3·15	3·17	3·19	3·21
1·7	3·23	3·25	3·27	3·29	3·31	3·33	3·36	3·38	3·40	3·42
1·8	3·44	3·46	3·48	3·50	3·52	3·54	3·56	3·58	3·60	3·62
1·9	3·64	3·66	3·68	3·70	3·72	3 75	3·77	3·79	3·81	3·83
2·0	3·85	3·87	3·89	3·91	3·93	3·96	3·98	4·00	4·02	4·04
2·1	4·06	4·08	4·10	4·12	4·14	4·17	4·19	4·21	4·23	4·25
2·2	4·27	4·29	4·31	4·33	4·35	4·38	4·40	4·42	4·44	4·46
2·3	4·48	4·50	4·52	4·55	4·57	4·59	4·61	4·64	4·66	4·68
2·4	4·70	4·72	4·74	4·77	4·79	4·81	4·83	4·86	4·88	4·90
2·5	4·92	4·94	4·96	4·99	5·01	5·03	5·05	5·08	5·10	5·12
2·6	5·14	5·16	5·18	5·21	5·23	5·25	5·27	5·30	5·32	5·34
2·7	5·36	5·39	5·41	5·43	5·45	5·47	5·49	5·52	5·54	5·56
2·8	5·59	5·61	5·63	5·66	5·68	5·70	5·72	5·74	5·77	5·79
2·9	5·81	5·83	5·85	5·88	5·90	5·92	5·94	5·97	5·99	6·01
3·0	6·04	6·06	6·08	6·11	6·13	6·15	6·17	6·20	6·22	6·24
3·1	6·27	6·29	6·31	6·34	6·36	6·38	6·40	6·43	6·45	6·47
3·2	6·50	6·52	6·54	6·57	6·59	6·62	6·64	6·66	6·68	6·70
3·3	6·73	6·75	6·77	6·80	6·82	6·85	6·87	6·89	6·92	6·94
3·4	6·97	6·99	7·01	7·04	7·07	7·09	7·12	7·14	7·16	7·18
3·5	7·20	7·22	7·25	7·28	7·31	7·33	7·35	7·38	7·41	7·43
3·6	7·45	7·47	7·49	7·52	7·54	7·57	7·59	7·61	7·64	7·66
3·7	7·69	7·71	7·74	7·76	7·79	7·81	7·83	7·86	7·88	7·91
3·8	7·94	7·96	7·99	8·01	8·03	8·06	8·08	8·11	8·14	8·17
3·9	8·19	8·22	8·25	8 27	8·29	8·31	8·34	8·36	8·38	8·41
4·0	8·43	8·46	8·48	8·51	8·53	8.56				

CREAM, 15 PER CENT. TO 45·60 PER CENT.

c.	0	1	2	3	4	5	6	7	8	9
	15·01	15·09	15·18	15·27	15·35	15·43	15·52	15·60	15·69	15·77
	15·86	15·94	16·03	16·11	16·20	16·29	16·37	16·46	16·55	16·64
	16·73	16·81	16·90	16·99	17·08	17·16	17·25	17·34	17·43	17·52
	17·60	17·69	17·78	17·87	17·95	18·04	18·13	18·22	18·31	18·40
	18·48	18·57	18·66	18·75	18·84	18·93	19·01	19·10	19·19	19·29
	19·37	19·46	19·55	19·64	19·73	19·82	19·91	20·00	20·09	20·17
	20·26	20·35	20·45	20·54	20·63	20·72	20·81	20·90	20·99	21·08
	21·17	21·26	21·36	21·45	21·54	21·63	21·72	21·81	21·90	22·00
	22·09	22·18	22·27	22·36	22·45	22·54	22·64	22·73	22·82	22·92
	23·01	23·10	23·20	23·29	23·38	23·47	23·56	23·66	23·75	23·85
	23·94	24·03	24·13	24·22	24·31	24·41	24·50	24·60	24·69	24·79
	24·88	24·98	25·07	25·17	25·26	25·35	25·45	25·54	25·64	25·73
	25·83	25·92	26·01	26·10	26·20	26·29	26·39	26·49	26·59	26·68
	26·77	26·87	26·97	27·07	27·17	27·26	27·36	27·46	27·55	27·65
	27·74	27·84	27·94	28·04	28·13	28·23	28·33	28·42	28·52	28·62
	28·72	28·81	28·91	29·00	29·10	29·20	29·30	29·40	29·50	29·60
	29·70	29·80	29·90	30·00	30·10	30·19	30·29	30·39	30·49	30·59
	30·69	30·79	30·89	30·99	31·09	31·19	31·29	31·39	31·49	31·59
	31·69	31·79	31·89	31·99	32·10	32·20	32·30	32·40	32·50	32·60
	32·71	32·81	32·91	33·01	33·11	33·21	33·31	33·41	33·51	33·61
	33·72	33·82	33·93	34·03	34·13	34·24	34·35	34·45	34·55	34·65
	34·75	34·85	34·96	35·06	35·16	35·27	35·38	35 48	35·59	35·69
	35·80	35·91	36·01	36·11	36·21	36·31	36·41	36·52	36·62	36·73
	36·83	36·94	37·04	37·15	37·25	37·36	37·47	37·57	37·68	37·78
	37·89	38·00	38·10	38·21	38·31	38·42	38·53	38·64	38·74	38·85
	38·96	39·07	39·18	39·28	39·39	39·50	39·61	39·71	39·82	39·93
	40·04	40·15	40·26	40·37	40·48	40·59	40·70	40·81	40·92	41·03
	41·14	41·25	41·36	41·47	41·58	41·69	41·80	41·91	42·02	42·13
	42·24	42·35	42·46	42·57	42·68	42·79	42·90	43·01	43·12	43·23
	43·34	43·45	43·57	43·68	43·79	43·90	44·02	44·13	44·25	44·36
	44·47	44·59	44·70	44·82	44·93	45·04	45·15	45·27	45·38	45·59
	45·60									

A SCHEME FOR THE SEPARATION AND DETECTION OF AL, CR, FE, CO, NI, MN, ZN, BA, CA, SR, MG, IN THE PRESENCE OF PHOSPHORIC, ARSENIC, OXALIC, BORIC, SILICIC, HYDROFLUORIC, ACETIC, TARTARIC ACIDS AND ORGANIC MATTER.

BY J. S. C. WELLS, PH.D., AND H. T. VULTE, PH.D.*

IT is a well-known fact that the usual separation with ammonium hydrate, chloride and sulphide fail in the presence of any of the above-mentioned acids. The various methods in use are long, and in most cases inaccurate. In presenting the following, we have tried to cover every point of importance, and wherever possible have used quantatitive methods for the sake of accuracy.

The scheme has been given a careful trial, and seems to meet all requirements.

If a solution containing the before-mentioned acids and bases is made alkaline with ammonium hydrate, the following is likely to occur :—

H_3PO_4	ppts.	Al	Cr	Fe(ic)	Co	Ni	Mn	Zn	Ba	Sr	Ca	Mg
H_3BO_3	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,
H_2SiO_3	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,
H F	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,	,,
$H_2C_2O_4$	,,	chiefly							,,	,,	,,	,,
H_3AsO_4	,,	Fe(ic) and Mg										
$H_2C_4H_4O_6$		prevent precipitation of Al, Cr, Fe, if boiling and neutral, $HC_2H_3O_2$ throws down Al, Cr and Fe as Basic Acetates.										
$HC_2H_3O_2$												
Organic Matter												

To test for the presence of these compounds three small portions of the main solution are taken as follows :—

Part I. Add HNO_3 and $(NH_4)_2 Mo O_4$ a yellow cryst. ppt. indicates phosphates, if heated gently a yellow colour or ppt. indicates silicates and arsenates.

Part II. Add $H_3C_4H_4O_6$ and $NH_4O H$ in excess a gelatinous ppt. indicates phosphates, borates, etc.

Part III. Add conc. H_2SO_4, and evaporate to dryness; a carbonaceous residue indicates organic acids or matter.

All the acids mentioned, except phosphoric, boric and arsenic, may be removed by evaporation with HNO_3 and ignition. Borates are soluble in a considerable excess of ammonium chloride, and give no trouble.

Arsenates are removed by reduction with SO_2 and precipitation as As_2S_3 by H_2S. This should be done before adding HNO_3.

After making these tests, return to main solution.

Boil out H_2S. Add a few drops HNO_3 (Note 1). If $H_2C_2O_4$ or organic matter is present evaporate to dryness and ignite gently. If not, evaporate, but do not ignite. Treat residue with HCl (conc.), dilute with H_2O and boil; it dissolves wholly or leaves a white residue of SiO_2. Filter. To filtrate add NH_4Cl and NH_4OH in slight excess, heat gently and filter quickly, wash ppt. A with hot water. To filtrate add $(NH_4)_2S$ slight xs heat, filter and wash ppt. B. Examine filtrate for the alkalies and alkaline earths. Transfer ppts. A and B to a porcelain dish and digest with a little $(NH_4)_2S$. (Note 2), filter and wash with H_2O and $(NH_4)_2 S$.

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* *School of Mines Quarterly*, X., 3.

<table>
<tr>
<td rowspan="7">.
$_4)_3PO_4$
$_4)_2S$
ect</td>
<td colspan="6">Residue $Al_2(OH)_6$ $Cr_2(OH)_6$ FeS, $Al_2(PO_4)_2$ $Cr_2(PO_4)_2$ $Ba_3(PO_4)_2$ $Sr_3(PO_4)_2$ $Ca_3(PO_4)_2$ NH_4 $Mg(PO_4)$, MnS, ZnS, CoS, NiS,S.
Treat with cold very dilute HCl sp. gr. 1·05, filter and wash.</td>
</tr>
<tr>
<td rowspan="6">Residue
NiS + CoS + S
Test a portion in Borax Bead
Blue = Co
Brown = Ni
If blue, diss. in Cl evap. to expel xs acids and Cl, dil. + H_2O nearly neutralise + KOH add KCY until ppt. formed is diss^{e}., boil for 4 or 5 minutes, cool, add large xs KOH + Br water a black ppt. $Ni_2(OH)_6$ wash with very dilute NH_4OH to remove last trace of Co test in Borax bead.
Brown Bead Ni</td>
<td colspan="5">Filtrate
Contains Fe, Al, Cr, Zn + Mn as chlorides, and Al, Cr, Ba, Sr, Ca, Mg as Phosphates.
Boil out H_2S, dilute and make alkaline with Na_2Co_3. add Bromine water and boil until Cr is all oxidised and Br. removed. Filter.</td>
</tr>
<tr>
<td rowspan="5">Filtrate
Na_2CrO_4
Acidify with $H\bar{A}$.
Test for Cr with $BaCl_2$ yellow ppt. $BaCrO_4$ = Cr. (Note 3.)</td>
<td colspan="4">Ppt. $Al_2(OH)_6$ $Al_2(PO_4)_2$ $Fe_2(OH)_6$ $Fe_2(PO_4)_2$ Ba, Sr, Ca, Mg Phos. Zn + Mn Basic Carbs. Diss. in least possible quantity of dil. HCl and add Fe_2Cl_6 until a few drops of the sol. give a yellow or red ppt. with NH_4OH. (Note 4.) Nearly neutralise with Na_2CO_3, dilute and add xs of $Na\bar{A}$. Boil and filter hot. Wash ppt. with boiling H_2O.</td>
</tr>
<tr>
<td colspan="2">Ppt.
Fe and Al as Phosphates and Basic Acetates add KOH and boil.</td>
<td colspan="2">Filtrate‖.
Zn Mn Ba Sr Ca Mg as acetates or chlorides add NH_4Cl NH_4OH + $(NH_4)_2S$. Filter and wash.</td>
</tr>
<tr>
<td rowspan="3">Ppt.
$Fe_2(OH)_6$
$Fe_2(PO_4)_2$</td>
<td rowspan="3">Filt.
Add HCl to acid reaction and then make alk. with $(NH_4)_2CO_3$ white ppt. $Al_2(OH)_6$ and $Al_2(PO_4)_2$.</td>
<td>Ppt.
ZnS + MnS.</td>
<td>Filtrate Ba, Ca, Sr, Mg acetates or chlorides. Examine in usual manner.</td>
</tr>
<tr>
<td colspan="2">Treat with $H\bar{A}$.</td>
</tr>
<tr>
<td>Res
ZnS
Treat on charcoal with $Co(NO_3)_2$ green mass Zn.</td>
<td>Solution
$Mn\bar{A}_2$ add KOH white ppt. turning brown in the air, fuse with Na_2Co_3. Green mass Na_2MnO_4 indicates Mn</td>
</tr>
</table>

NOTES.

I. HNO_3 is added to oxidize Fe and destroy $H_2C_2O_4$, organic acids and matter.

II. NH_4OH ppts. Fe, Al, Cr as hydrates and phosphates, and the other metals of the group as phosphates, if they are in combination with that acid, it also ppts. all of the phosphates of the alkaline earths that may be present.

Ppt. A is treated with $(NH_4)_2S$ to convert the phosphates (except those of Al, Cr, Ba, Sr, Ca and Mg) into sulphides, the H_3PO_4 is taken up by the NH_4OH in a soluble form and filtered off, thereby making a partial separation of the phosphoric acid.

III. A pink or green colouration may be caused by the oxidation of Mn to permanganate or manganate, neither interferes with the $BaCl_2$ test.

IV. Ferric salt is added to carry down the phosphoric acid, as $Fe_2(PO_4)_2$, white solution must be alkaline with NH_4OH, when all the H_3PO_4 is pptd. the NH_4OH brings down the yellow or brown $Fe_2(OH)_6$.

V. Instead of scheme as above the following may be used:—

Filt. containing Mn, Zn, Ba, Sr, Ca, Mg as acetates or chlorides add Br and boil as long as a black ppt. is formed and colour of Br remains; filter.

<table>
<tr><td rowspan="2">Ppt.
MnO_2H_2O
test in
Na_2CO_3
bead.</td><td colspan="2">Filt. Zn, Ba, Sr, Ca, Mg acetates or chlorides.
Warm, pass a rapid current of H_2S, filter.</td></tr>
<tr><td>ZnS
white, if
dark coloured
test on charcoal with
$Co(NO_3)_2$.</td><td>Filt.
Boil out H_2S, and proceed as usual.</td></tr>
</table>

ON THE DENSITIES AND REFRACTIVE INDICES OF CERTAIN OILS.

By J. H. Long.

(Continued from page 178.)

Mustard Oil.

The oil of black mustard may be classed among the cheaper oils, and when properly refined has numerous uses. I have examined two samples of known purity, and a third prepared from one of these by partial saponification. The first two had been well refined, and were almost entirely free from the odour of the volatile oil.

No. 2. A sample obtained in Chicago.

T.	D.	T.	N.
4·0°	·9222	14·5°	1·4769
19·1	·9122	17·5	1·4751
34·0	·9026	20·5	1·4739
48·6	·8932	22·5	1·4732
		24·5	1·4724
		28·9	1·4706
		35·0	1·4683
		40·0	1·4663

No. 15. Oil refined from the above by treatment with a 1 per cent. solution of potassium hydrate.

T.	D.	T.	N.
1·5°	·9251	14·8°	1·4771
16·6	·9149	19·3	1·4749
33·6	·9037	21·3	1·4739
47·4	·8946	23·3	1·4729
		25·8	1·4720
		31·3	1·4700
		34·3	1·4688
		37·3	1·4677

No. 17. A sample obtained from a New York importer.

T.	D.	T.	N.
1·7°	·9239	15·3°	1·4759
18·6	·9128	19·3	1·4743
36·0	·9014	21·3	1·4736
45·8	·8948	23·8	1·4726
		27·3	1·4712
		31·8	1·4694
		36·8	1·4674
		39·3	1·4665

The temperature coefficients are nearly as observed in the other oils.

Peanut Oil.

One sample of oil, partially refined, was examined, with these results.

No. 19.

T.	D.	T.	N.
3·2°	·9309	15·8°	1·4736
17·9	·9186	19·8	1·4718
34·4	·9074	23·3	1·4704
47·7	·8984	25·3	1·4696
		28·3	1·4684
		34·3	1·4661
		39·2	1·4641

While the change in the density is comparatively rapid between the lowest tem- ratures, it amounts to ·00068 for each degree between the mean temperatures, that is, e rate is about as before given. The variation in the refractive index is normal.

Castor Oil.

The following results were obtained by examination of a sample of well-refined d-pressed oil.

No. 3.

T.	D.	T.	N.
4·0°	·9695	14·4°	1·4831
7·1	·9675	18·4	1·4806
18·0	·9602	20·4	1·4790
32·5	·9505	22·9	1·4781
46·8	·9410	25·4	1·4771
		28·4	1·4759
		33·9	1·4741
		37·3	1·4728

Lard Oil.

A sample of pure oil, pressed in Chicago, was examined, and gave these figures. As ecame thick at the lowest temperature, the density determination at that point is ably not quite accurate.

No. 13.

T.	D.	T.	N.
1·8°	·9287	20·3°	1·4685
15·5	·9154	24·3	1·4669
32·5	·9036	27·3	1·4657
48·5	·8926	29·3	1·4649
		32·3	1·4637
		36·3	1·4622
		39·2	1·4612

In order to compare the values for the different oils with each other, as well as with similar data obtained by other observers, I have calculated from the above direct observations the numbers in the tables below. In most cases the curve representing the variations in specific gravity is nearly a straight line, and without introducing appreciable error it can be taken as linear between the intermediate temperatures. Assuming these points as correctly determined, I have interpolated values from 18° to 25° and for 30° and 35°.

By means of carefully drawn curves I have interpolated, from the direct determinations of N, the values for the same degrees of temperature. The numbers thus obtained are undoubtedly as accurate and satisfactory as could be obtained by a laborious method of calculation. Finally, I have calculated the "specific refractive energy," $\frac{N-1}{D}$, and give these values in another column.

(To be continued.)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

A SOURCE OF ERROR IN THE SEPARATION OF A SMALL QUANTITY OF MANGANESE FROM A LARGE AMOUNT OF LIME BY AMMONIUM SULPHIDE. BY L. BLUM. (*Zeits. f. Anal. Chem.* 28, 454.)—In the precipitation of manganese by ammonium sulphide, Fresenius recommends that in all cases at least twenty-four hours, and in the presence of very small quantities of manganese, forty-eight hours should elapse before filtration. The author finds that with much lime and little manganese, a crystalline precipitate of calcium thiosulphate is deposited; to avoid this he filters off at once after a good boiling up. H. D. R.

THE ESTIMATION OF NITRIC NITROGEN BY KJELDAHL'S METHOD. BY DR OTTO FOERSTER (*Zeits. f. Anal. Chem.* 28, 422).—By the application of phenol-sulphonic acid to the estimation of nitrogen by Kjeldahl's method in nitrates, the results are obtained slightly low, because on heating some of the nitric acid volatilises without having combined with the phenol. This loss may be prevented by the use of a second substance, forming a less volatile combination with the nitric acid. The author has made many experiments with a phenol-sulphonic acid solution, containing varying quantities of phenol, both with and without reducing agent, preferrably sodium thiosulphate, which with the nitric and sulphuric acids forms "Lead Chamber Crystals." Another source of error is due to the use of india-rubber connexions, there being a loss in his experiment of 2 m. grs. of nitrogen for each gram. of nitre. This is avoided by the use of an apparatus containing the standard acid, consisting of 5 bulbs (described and figured in Fresenius' "Quantitative Analysis," 6th edition, 1. 224), into which the tube from the distillation flask is directly led. With this apparatus, and by the use of a phenol-sulphonic acid solution containing 5-6 per cent. phenol, of which 30 c.c. are used for each grm. of nitre, and by the addition of about 1 grm. sodium thiosulphate, the author finds results with potassium nitrate very closely agreeing with the theory. H. D. R.

THE ESTIMATION OF THE EXTRACT IN WINE BY THE INDIRECT METHOD. BY E EGGER (*Zeits. f. Anal. Chem.* 28, 396).—A commission appointed in 1874 to examine the methods of Wine Analysis, give the following instruction for taking the "extract": "50 c.c. of wine, measured at 15° C. are evaporated in a platinum basin (80 m.m. across, 50 m.m. high, holding 75 c.c., and weighing about 20 grms.) on a water bath, and the residue dried for two and a half hours in a water oven. Of wines rich in sugar (*i.e.*, containing ·5 grm. sugar in 100 c.c.), a quantity suitably diluted is taken, so that from 1-1·5 grms. are left."

Both Schultze (*Zeits. f. Anal. Chem.* 19, 104) and Hager (*ibid.* 17, 502) give tables for the calculation of the extract from the density of the wine from which the alcohol has been boiled off.

The author has compared in 150 experiments the results obtained directly with those calculated from both tables, with the following results:—

Difference between numbers calculated and those found.	Schultze.	Hager.
under ·2 per cent.	in 132 experiments.	in 6 experiments.
·2 — ·5 "	18 "	115 "
over ·5 "	— "	29 "

He therefore gives the preference to Schultze's tables; the low results given by the uses of Hager's figures being due to the fact that he dried the malt extract (which he used in the preparation of the solutions from which his tables were calculated) at 110° C.

H. D. R.

THE ESTIMATION OF NITROGEN BY KJELDAHL'S METHOD. BY F. MARTINOTTI (*Zeits. f. Anal. Chem.* 28, 415).—From comparative analyses with manures, the author concludes that in the absence of nitrates the Kjeldahl method is preferrable to the soda-lime method. He considers the use of mercury, and in many cases potassium permanganate, unnecessary, and finds that in their absence the distillation is more regular. In the presence of nitrates he does not recommend the use of the Kjeldahl-Jodlbauer method, but prefers to first reduce the nitrate by heating with concentrated hydrochloric acid and ferrous chloride.

H. D. R.

PURIFICATION OF LITMUS. BY DR. OTTO FOERSTER (*Zeits. f. Anal. Chem.* 28, 428).—Dr. Foerster recommends, as an indicator in Kjeldahl's method, a solution of 9 grms. litmus and 1 grm. malachite green in 1 litre of alcohol. Commercial litmus is extracted with ordinary alcohol in the cold, then digested with water, the solution filtered and evaporated; the residue is again dissolved in water, filtered, and the filtrate precipitated with absolute alcohol, to which some acetic acid is added. The precipitate is collected on a filter and washed with alcohol, whereby a violet, fluorescent colouring matter is separated; the residue is again dissolved in water and precipitated, filtered, and washed as before. By the second repetition only a small quantity of the colouring matter is separated. The precipitate is stirred up with alcohol to remove the acetic acid, and then again dissolved in water and the solution filtered. The colouring matter finally precipitated with slightly ammonical alcohol, collected on a filter, washed with strong alcohol and dried, is used to make up the solution.

H. D. R.

Purity of Quinine Sulphate. J. E. De Vry. *Nederl. Tydschr v. Pharmacie*, etc. —1. The chromate test; two grammes of quinine sulphate are dissolved in 80 c.c. of boiling water and mixed with a solution of ·55 grammes of potassic chromate. After the liquid has cooled down until 15°C., the liquid is filtered off from the precipitated quinine chromate. Solution of caustic soda is now added until the liquid is distinctly alkaline to phenol-phthalein paper. If the sample is practically pure, viz., contains no more than ·5 per cent. of other alkaloids, the liquid will remain clear; but if large amounts of impurities are present, the liquid will get more or less turbid. Although the process does not lay claim to *quantitative* accuracy, still, with a little practice, a fair idea may be got about the percentage of impurities. The quinine may be readily recovered from the collected chromate by dissolving the mass in boiling water and precipitating with soda ley. 2. The optical test; two grammes of the sample are dissolved in 80 c.c. of hot water and mixed with a hot solution of two grammes of Rochelle salts. After a little while, the tartrate begins to crystallize out. After standing for a day, the precipitate is filtered off, washed and air dried. The molecular rotation of the tartrate $[A_D]$ 0 of a sample submitted to the author was 209·33, showing it to consist of 92·34 QT and 7·56 C^dT, which, calculated on original sample, corresponds with 92·62 quinine sulphate and 7·38 cinchonidine ditto. If, as sometimes happens, the sample has an alkaline reaction, it must first be neutralised with dilute sulphuric acid; otherwise a basic tartrate will be obtained, which gives too low a polarisation. For the detection of large quantities of cinchonidine, the ether test answers well enough, providing the ether is absolutely free from alcohol. A sample from a well known French firm was guaranteed not to give any separation on agitating with alkali and ether. The author really found such to be the case when he used common ether containing alcohol, but by using pure ether the impurity was at once detected. L. de K.

New Test for Antiarine. Wefers Bettink. *Nederl. Tydschr. v. Pharmacie* etc., April, 1889.—The author discovered a delicate reaction for this formidable arrow poison. If 1 c.c. of a 5 per cent. solution of sodium carbonate is boiled with three drops of a cold saturated solution of picric acid, the yellow colour does not perceptibly change. If, however, as little as ·0001 gramme of antiarine is added, the colour turns orange-red. The colour is not produced by such glucosides as aesculine, amygdaline, phloroglucine, digitaline, and picrotoxine. This test, in conjunction with physiological experiments, enables us to recognise the poison. A few milligrammes of antiarine, when injected in the blood, will kill dogs or rabbits in a few seconds.

L. de K.

Estimation of Cantharidin. J. B. Nagelvoort. *Nederl. Tydschr. v. Pharmacie* etc., July, 1889.—The best method as yet known, originated by Greenish, gives certainly fair results in experienced hands, but, it cannot be denied it is a most troublesome and costly process. For instance, 10 grammes of the cantharides must be exhausted with benzol to remove the fat, and frequently as much as 200 c.c. of this fluid will be required. The exhausted residue must be digested in soda ley, and then dried, when it will be often found almost impossible to powder. The cantharadin is dissolved out by her and chloroform, and finally purified by alcohol. Now, as it is very perceptibly luble in benzol and in alcohol, the volumes of these fluids must be carefully noted and rrections must be applied, so that sometimes one half of the cantharidin found is made by allowances. The author now proposes the following process: 10 grammes of the ntharides are moistened with a 10 per cent. soda ley, and put in a warm place for six urs. The mass is now acidified with hydrochloric acid, transferred to a Soxhlet's tube d fully exhausted by means of chloroform (about 50 c.c.). After evaporating off the loroform, the residue is freed from fat by means of carbon disulphide, and then re-issolved in chloroform. The liquid is filtered off and evaporated to dryness at 79·5° C., hen the cantharidin may be weighed.

L. DE K.

REVIEWS.

THE CHEMICAL ANALYSIS OF IRON: A COMPLETE ACCOUNT OF ALL THE BEST KNOWN METHODS FOR THE ANALYSIS OF IRON, STEEL, PIG-IRON, IRON ORE, LIMESTONE, SLAG, CLAY, SAND, COAL, COKE, AND FURNACE AND PRODUCER GASES. By ANDREW ALEXANDER BLAIR. London: Whittaker and Co., White Hart Street, Paternoster Square.

THE immense mass of matter which has appeared on the subject of the analysis of iron and of the various materials employed in its manufacture, or produced in the course thereof, has been so widely distributed through all sorts of periodicals, that a good compilation like that now under consideration is really a most serviceable book. As might have been expected from the author's antecedents—he having (after graduating in the United States Naval Academy in 1866) been chief chemist to the States Board appointed to test iron, steel, and other metals in 1875, and subsequently chief chemist to the United States Geological Survey—the work is something more than a mere compilation. With very few exceptions the descriptions are the results of the author's own experience in the use of the particular methods detailed, while the points specially insisted upon are those that seemed to him to be specially important when practically performing the processes in question. In the preface the author seems to indicate that "works" chemists are not better treated in America than they are with us, because he says:—"Many of the special forms of apparatus are of my own contrivance; they have proved extremely useful to me, and I hope may facilitate in some cases the work of iron chemists, to whom often very little is given and of whom very much is required." Like most of the American books that have lately appeared, the style both of printing and illustrations are admirable; the former is very easy to read, and the latter are plain and sufficiently numerous; we should be inclined to think that there will be very few analysts interested in the subject that will not provide themselves with a copy of the book. It is not, however, to the specialist only that the work will be useful, because it is so shortly and yet lucidly written as to be just the sort of book that a general chemist would keep handy in the event of his being called upon to engage in such analysis; when it would certainly come in very useful and save much loss of time in hunting through one's library.

A Treatise on Manures; or, The Philosophy of Manuring: A Practical Handbook for the Agriculturist, Manufacturer, and Student. By A. B. Griffiths, Ph.D., F.R.S.E., etc. London: Whittaker and Co., White Hart Street, Paternoster Square.

This is one of a set of handy books now being published by Messrs. Whittaker under the name of the "Specialist's Series." It may be at once said that it meets a decided want. It is lucidly written, and at the same time is sufficiently popular to be appreciated by the class of men to whom it ought to be most vitally interesting, viz., the farmers. It is only to be hoped that they will, as a body, carefully peruse it; but, in his preface, the author does not seem to be very sanguine on this point, because he says:—"Farmers as a class hold most tenaciously to their old customs, and are most jealous of innovations. They remember the days of prosperity, and, hoping for their return, ignore the immense advance in every other industry, and continue in the old routine to compete with the foreign agriculturist in the production of cereals, while enormous quantities of commodities are imported that could be more advantageously produced by themselves. In the words of an anonymous writer in a weekly paper: 'Orthodoxy is an institution of a past generation, and no more adapted to the present times than her contemporaries the flail or the spinning-wheel. She cannot even be defended on the ground of continuing to supply a required need in the absence of something better. She stands proved a complete failure, and, as such, should be blotted out. She impedes progress; she insists on the cultivation of crops that of foreknowledge will result in loss; and she admits of no departure to suit existing circumstances. . . . Can nothing be said in favour of Orthodoxy? Is there no favourable aspect? No, not one! She is neither the friend of the landlord, tenant, nor labourer. Many of the natural advantages which we formerly possessed as a nation have been taken from us by that great leveller, Steam, which brings products to our shores and equalises the advantages which one nation formerly enjoyed over another. We cannot retrograde to the blissful past; therefore the farmer must alter his system of sowing, manuring, etc., to suit the changed condition of things. What is the use of still continuing a system that does not pay? Self-interest, therefore, should lead farmers to throw away their old prejudices, and listen more attentively to the teaching of science rather than look for help to politicians.'"

The book begins with a short historical account of the progress of agriculture, and then takes up the constituents of plants and the physiology of their nutrition. In urging the necessity of manuring, the author introduces a pungent little illustration by telling the story of the priest, who, having been called to pray over the barren fields of his parishioners, and having passed from one enclosure to another, at the same time pronouncing his benediction over each, came to a most unpromising case. He surveyed the sterile acres in despair. "Ah," said he, "brethren, no use to pray here—this needs manure." While, however, thus urging the use of manures, the author carefully distinguishes between their use and abuse, because we find him quoting the following valuable words of Stutzer:—"The manure produced on the farm is in very few cases sufficient to supply the cultivated plant with the nitrogen it requires to produce crops securing the highest possible clear profit. The practical farmer must therefore have recourse to artificial manure . . . . Even with this high farming, there are fields in which

s may be brought to a richer development by an increase of manure, and yet
ased manuring is not advisable as soon as it does not pay. It is not the aim
rmer to produce the greatest crops irrespective of profit or loss, but to realise
st clear profit; therefore more manure must not be used than is profitable."
ing discussed all the varieties of natural and artificial manures, and gone
ly into the constituents and modes of manufacture of the latter, the author
pecial study of the manurial value of ferrous sulphate, a subject with which
has been previously identified. Then follows a chapter upon Ville's system of
, and the whole ends with directions for the analysis and valuation of fertilizers.
no doubt that this work is one of the best that has yet been produced at a
price, so as to be within the reach of all.

ION OF WATER FOR SANITARY AND TECHNICAL PURPOSES. By HENRY LEFFMANN,
., and WILLIAM BEAM, M.A. Philadelphia: P. Blakiston, Son and Co.

e book contains within 100 pages full instructions for the sanitary and
ive analysis of water, and as such, it can, of course, be little more than a re-
n of well-known matter. It is probable that such a publication may fill a void
an chemical literature, but, over here, there is really very little opening for the
because the ground is already fully covered by Wanklyn, Fox, and many others.
al quantitative part is based on Fresenius, and we suppose that a better model
have been chosen. The sanitary part is, with some slight deviations, practically
uctions for water analysis approved by the Society of Public Analysts, and
in our columns some years ago. The two main points of difference consist in
ess estimation and in the nitrates and nitrites. In the former case the time-
soap test is given up, and Mr. Hehner's method (with sodium carbonate and
acid) is substituted. Whether this is a good departure or not, it will certainly
ted to a good deal of conservative criticism. In the latter case all the usual
in common use are given up, and the methods recommended are the purely
ic ones first brought into notice (if we are not mistaken) in our columns by
Smith. Here we think there is the decided advantage that the authors
eparate estimation of the nitrogen as nitrates and that as nitrites, and so un-
gain more information as to the actual stage of oxidation. As these processes
r obtained a firm hold in this country, it will be interesting to some of our
extract them here, and thus give an idea of the general style of the book.

NITROGEN AS NITRATES.

REQUIRED :—

Phenyl Sulphate.—18·5 c.c. of strong sulphuric acid are added to 1·5 c.c. of
3 grams. of pure phenol. Preserve in a tightly-stoppered bottle.

dard Potassium Nitrate.—0·722 gram. of potassium nitrate, previously heated
erature just sufficient to fuse it, is dissolved in water, and the solution made up
.c. 1 c.c. of this solution will contain ·0001 grm. of nitrogen.

ANALYTICAL PROCESS :—

A measured volume of the water is evaporated just to dryness in a platinum or porcelain basin. 1 c.c. of the acid phenyl sulphate is added and thoroughly mixed with the residue by means of a glass rod. 1 c.c. of water is added, three drops of strong sulphuric acid, and the dish gently warmed. The liquid is then diluted with about 25 c.c. of water, ammonium hydroxide added in excess, and the solution made up to 100 c.c.

The reactions are :—

$$\underset{}{\overset{\text{Acid phenyl sulphate.}}{HC_6H_5SO_4}} + 3HNO_3 = \overset{\text{Trinitrophenol (picric acid).}}{HC_6H_2(NO_2)_3O} + H_2SO_4 + 2H_2O.$$

$$HC_6H_2(NO_2)_3O + NH_4HO = \overset{\text{Ammonium picrate.}}{NH_4C_6H_2(NO_2)_3O} + H_2O.$$

The ammonium picrate imparts to the solution a yellow colour, the intensity of which is proportional to the amount present.

Five c.c. of the standard solution of potassium nitrate is now similarly evaporated in a platinum basin treated as above, and made up to 100 c.c. The colour produced is compared to that given by the water; and one or the other of the solutions diluted until the tints of the two agree. The comparative volumes of the liquids furnish the necessary data for determining the amount of nitrate present, as the following example will show :—

Five c.c. of standard nitrate is treated as above, and made up to 100 c.c.

$$\begin{array}{rl} \cdot 0001 & \\ 5 & \\ \hline \cdot 0005 & \text{gram. N per 100 c.c.} \\ 10 & \\ \hline \cdot 0050 & \text{,, ,, 1,000 ,,} \end{array}$$

Suppose 100 c.c. water similarly treated is found to require dilution to 150 c.c. before the tint will match that of the standard; then

$$100 : 150 :: \cdot 005 : \cdot 0075$$

i.e., water contains 7·5 milligrams. of nitrogen as NO_3 per litre.

The ammonium picrate solution keeps very well, especially in the dark. A good plan, therefore, is to make up a standard solution equivalent to, say, ten milligrams. of nitrogen as nitrate per litre, to which the colour obtained from the water may be directly compared.

The results obtained by this method are quite accurate. Care should be taken that the same quantity of acid phenyl sulphate is used for the water and for the comparison liquid, otherwise different tints instead of depths of tints are produced.

With subsoil and other waters probably containing much nitrates, 10 c.c. of the sample will be sufficient for the test, but with river and spring waters, 25 to 100 c.c. may be used. When the organic matter is sufficient to colour the residue, it will be well to purify the water by addition of alum and subsequent filtration, before evaporating.

NITROGEN AS NITRITES.

SOLUTIONS REQUIRED :—

Naphthylammonium Chloride.—Saturated solution in water free from nitrites. It should be colourless; a small quantity of animal charcoal allowed to remain in the bottle will keep it in this condition.

'ara-amido-benzene-sulphonic Acid [*Sulphanilic Acid*].—Saturated solution in water om nitrites.

Iydrochloric Acid.—25 c.c. of concentrated pure hydrochloric acid added to 75 c.c. free from nitrites.

tandard Sodium Nitrite.—0·275 gram. pure silver nitrite is dissolved in pure and a dilute solution of pure sodium chloride added until the precipitate ceases to It is then diluted with pure water to 250 c.c., and allowed to stand until clear. e 10 c.c. of this solution are diluted to 100 c.c. It is to be kept in the dark.

ne c.c. of the dilute solution is equivalent to ·00001 gram. nitrogen.

he silver nitrite is prepared thus: A hot concentrated solution of silver nitrate is to a concentrated solution of the purest sodium or potassium nitrite available, l while hot and allowed to cool. The silver nitrite will separate in fine needle- ystals, which are freed from the mother liquor by filtration by the aid of a filter- The crystals are dissolved in the smallest possible quantity of hot water, allowed and again separated by means of the pump. They are then thoroughly dried in ter-bath, and preserved in a tightly-stoppered bottle away from the light. Their may be tested by heating a weighed quantity to redness in a tarred porcelain le and noting the weight of the metallic silver. 154 parts of $AgNO_2$ leave a e of 108 parts Ag.

TICAL PROCESS :—

00 c.c. of the water is placed in one of the colour-comparison cylinders, the ring vessel and cylinder having previously been rinsed with the water to be tested. ans of a pipette, one c.c. each of the solutions of sulphanilic acid, dilute hydro- acid and naphthylammonium chloride is dropped into the water in the order l. It is convenient to have three pipettes for this test, and to use them for no purpose. In any case, the pipette must be rinsed out thoroughly with nitrite- ater each time before using, as nitrites in quantity sufficient to give a distinct n may be taken up from the air.

ne c.c. of the standard nitrite solution is placed in another clean cylinder, made th nitrite-free water to 100 c.c. and treated with the reagents as above.

n the presence of nitrites a pink colour is produced, which in dilute solutions may e half an hour for complete development. At the end of this time the two ns are compared, the colours equalised by diluting the darker, and the calculation as explained under the estimation of nitrates.

he following are the reactions :—

Para-amido-benzene-sulphonic acid. Nitrous acid. Para-diazo-benzene-sulphonic acid.

$$C_6H_4NH_2HSO_3 + HNO_2 = C_6H_4N_2SO_3 + 2H_2O$$

Naphthylammonium chloride. Azo-alpha-amido-naphthalene-parazo-benzene-sulphonic acid.

$$C_6H_4N_2SO_3 + C_{10}H_7NH_3Cl = C_{10}H_6(NH_2)NNC_6H_4HSO_3 + HCl$$

he last-named body gives the colour to the liquid.

his extract will show that the book is really well written, and will be found very to those whose libraries are not already glutted with such literature.

Commercial Organic Analysis: A Treatise on the Properties, Proximate Analytical Examination, and Modes of Assaying the Various Organic Chemicals and Products Employed in the Arts, Manufactures, Medicine, etc.; with Concise Methods for the Detection and Determination of their Impurities, Adulterations, and Products of Decomposition. By Alfred H. Allen, F.I.C., F.C.S. Second edition, revised and enlarged. Volume III., Part I. London: J. and A. Churchill, New Burlington Street.

The new volume of Mr. Allen's work, although waited for by most of the profession with considerable impatience, amply justifies the care bestowed on it by the author, and simply goes to prove that what is done deliberately is generally done well. The volume now before us is devoted to the consideration of the acid derivatives of the phenols, aromatic acids, tannins, dyes and colouring matters. To attempt anything like an exhaustive criticism of such a work within the space available in our columns would be to do justice neither to the book nor to our readers. No doubt by this time most of them have already added the volume to their libraries, and all we can say is that, if any have not yet done so, then the sooner they get it the better, because it is simply an unrivalled compilation of all the most recent matter that has appeared on the subject on which it treats. The author is nothing if not having a shot at somebody, and he is especially in his element when he can get at the pharmacopœia compilers. In this volume he has two capital chances over zinc sulphocarbolate and chrysophanic acid respectively. The chapter on tannins is very complete, many excellent tables of reactions, etc., being given, but the great bulk of the book is occupied by the dyes. The analytical instructions and tables for the recognition of the dye that has been used on any fabric, etc., extend over fifty pages, and no one can look at this part of the work without being forcibly struck with the enormous advance in analytical chemistry that has taken place within the present generation, and the rapidly approaching impossibility of any man saying, "I know chemistry." As years roll on chemists are driven to specialise and to devote themselves to some particular branch of analysis, and we venture to predict that the old general analytical chemist, who took up any analysis with a light heart, will soon become an extinct animal. If anything could work to help a man to be a sort of Admirable Crichton in analysis, it is the production of such books as this. Mr. Allen is to be congratulated on the continued successful progress of his heavy undertaking. From the advance in all organic work he will no sooner have issued Vol. III., Part 2, than he will have to start again on the revise of Vol. I. We fear that, to an author of such books, life must become at last something very nearly similar to the existence experienced by Mr. Mantalini of well-known celebrity for the terse way in which he expressed his feelings on the subject.

PRIZE OFFERED FOR A PROCESS OF BUTTER ANALYSIS.

The Royal Society of Science in Denmark has offered a prize of Kr. 600 for a chemical investigation as to the fatty acids present in butter-fat, each acid to be isolated and sufficiently identified. The relative amount of the different fatty acids, of which the investigation is expected to give information, should be given with the methods employed, as far as they are new or otherwise of interest, and especially is wanted the proportion between oleic acid and palmetic acid with its higher homologues.

Papers, which may be written in Danish, Swedish, English, German, French, or Latin, are to bear no name but a motto, and a sealed envelope with the same motto, enclosing the name, position, and address of the author, must be sent with the paper, which should be sent before the 31st of October, 1890, to Dr. H. G. Zeuthen, Secretary of the Society.

THE ANALYST.

NOVEMBER, 1889.

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ON THE ESTIMATION OF SULPHUR IN PYRITES.

By G. Lunge.*

l-known fact that the estimation of sulphuric acid as barium sulphate, is not rate when the solution contains iron salts. Although the precipitate is always oloured by iron, the results are, however, slightly too low. The reason of ar fact has been completely cleared up by the investigation of Jannasch and it is caused by the formation of an insoluble barium—iron sulphate. These herefore, condemn in their paper my process for the estimation of sulphur in being inaccurate, but they do not mention that this can only refer to an old mine, published in my work on the soda industry, as already in 1881 I advised ecipitate the iron with ammonia. I now hear that Jannasch finds this to be y reliable method. Looking at the immense importance of the process r perhaps a million tons of the article are sold on analytical certificates), I had ommenced a thorough investigation, so as to make everything perfectly t us first see, however, how matters stood before Jannasch's and Richards' on.

y work on soda manufacturing, I have preferred to recommend the treatment of s with aqua regia, instead of the fusion with nitrate and carbonate of soda.

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* *Zeitschr. f. angew Chemie, No.* 17, 89.

Fresenius, who made his assistants perform a number of analyses, however, showed th dry process to give better results, on account of the complete isolation of the iron. One of his assistants got even 1 per cent. of sulphur too low, which was attributed to the solvent action of the ferric chloride on the barium sulphate.

Now I found in 1881, first, that the presence of iron, as ferric chloride, renders the precipitation with barium chloride somewhat inaccurate, as the barium sulphate is always ferruginous, and can only be freed from iron by using large quantities of hydrochloric acid, which causes a loss of about ·5 per cent. of barium sulphate. In two analyses the percentage of sulphur was found to be from 46·7 to 47·32 per cent.; average 46·98 per cent. I further proved that perfectly concordant and accurate results may be obtained by precipitating the iron with the smallest possible amount of ammonia, and washing the precipitate on a filter with boiling water. In all my experiments I tested for sulphuric acid in the ferric hydrate, by fusing with soda, and dissolving the mass in water, etc., but I have never found a trace of it, so it is a needless operation.

Then I showed that the second method gives a little more sulphur than the first, but after all, only about ± ·18 per cent. How they managed to get as much as 1 per cent. difference in Fresenius' laboratory, I think I can explain by their pyrites containing traces of galena, heavy spar, or even gypsum.

In some experiments by Jannasch and Richards, there could be no question of galena or heavy spar, but, as they observed, a prolonged ignition of the precipitate may cause volatilisation of some of the sulphate, and any increase in weight through presence of iron be more than counteracted through this loss. The problem to find a reliable process for sulphur estimation by a wet process, was sufficiently solved by my labours. The process has been generally introduced in Germany and England, and is the only accepted process in the German soda trade, and the many complaints, of analysts differing in their results (by using the older process), have gradually died out. I now wish to show that Jannasch's and Richards' objections only relate to my first published method. They describe twenty-five different experiments, which all again prove the already known fact that in presence of iron the estimation of sulphur as barium sulphate is incorrect. Their last experiment also *qualitatively* shows the formerly not known cause of this error, viz., the formation of an insoluble barium-iron sulphate, which, on ignition, slowly but gradually loses the part of the sulphuric anhydride which may be supposed to be in combination with the iron. They say: The experiment (heating the precipitate at a bright-red heat) can be continued for half an hour without getting rid of the fumes of sulphuric anhydride, and the precipitate is constantly increasing in colour. Whilst they got from 10 c.c. of their normal acid 1·1608 gram. barium sulphate in absence of iron, when this was added they got an average of only 1·117, or about 4 per cent. too little. These remarkably large differences, which far exceed those got by Fresenius, no doubt were caused by the unusual and continued ignition. However, their experiments once more conclusively show that the estimation of the sulphur in presence of iron is not reliable, and regularly gives results too low, although the loss must vary according to circumstances. But this does not occur when the iron is first removed by means of ammonia, which process is, however, not mentioned in any of their twenty-five experiments. True, they casually mention that the previous precipitation of the by ammonia is inadmissible, since even after a double precipitation the precipitate retains ·5 per cent of sulphuric acid (as basic ferric sulphate). They do not, how- r, seem to have made any accurate experiments themselves. We will also presently that it really depends on how the precipitation is performed, and that if sufficient care ot taken, even a far greater quantity than ·5 per cent. will be retained by the precipitate. t, as already mentioned, I took care not to commit any errors, and have convinced self of the absolute absence of sulphuric acid from the precipitate. Now all this ht to be a sufficient answer to those who think my process inaccurate, but the matter ng of such vast analytical importance, I resolved to again investigate the matter from inning to end. As, however, my many professional duties prevented me from doing analyses myself, I have had them performed by two gentlemen in whose abilities I ce the greatest confidence, and who have investigated the matter quite independently each other.

Ch. Barbezat prepared (as I did in 1881) a solution which represented an iron itis, viz., contained 2 molecules of sulphuric acid for 1 atom of iron. The solution not only pipetted off, but also weighed. The liquids were precipitated by ammonia, ferric hydrate washed with boiling water, and, after drying, fused on the blow-pipe h soda. After dissolving in water and filtering, hydrochloric acid and barium chloride added, and the fluid allowed to stand a long time. Now a remarkable fact was closed. In his first two experiments, Barbezat brought the iron sulphate solution to ling heat, added ammonia till it had a faint smell, boiled until smell had disappeared, then collected and washed on to a filter, until the filtrate measured nearly 700 c.c. dried precipitate gave, after fusion with soda, a very distinct reaction for sulphuric d, which, when weighed, amounted to 3·6 per cent. of the total, or a still larger error n got by Jannasch and Richards, who only found ·5 per cent.

Now, in my description of the process, I have particularly laid stress on using a derate *excess* of ammonia. I said : "The moderate hot fluid is mixed with a not too ge excess of ammonia, filtered off after about ten minutes, and washed on the filter h boiling water until the filtrate gives no longer a cloud with barium chloride, even er some time." His attention being called to this, Barbezat repeated the experiments, now found in three cases *not a trace*, and in another case only the merest sign of phuric acid, and this trace was most likely caused by absorption of sulphurous pour during the fusion.

The experiments conducted by Anast Obregia are more to the point. He worked h iron pyritis itself, which he had obtained from Spain, and which was free from per, lead, and other sulphides. From the finely agated sample, 1 gramme was en for analysis. Experiments were made (*a*) with my old process, without separa- of iron; (*b*) with my modified process of 1881; (*c*) with Fresenius' process, the on with soda and nitre. (*a*) and (*b*) were heated exactly according to my directions h aqua regia, consisting of one part of fuming hydrochloric acid and three parts of ic acid of 1·4 sp. gr., both, of course, free from sulphate. The evaporation on the er-bath takes about fifteen minutes, and no free sulphur separates. When dry, the is treated twice with excess of hydrochloric acid, and again evaporated. 100 c.c.

of water and four drops of hydrochloric acid are then added, and the liquid filtered from any gangue.

Three experiments (*a*) were performed. The boiling fluid was directly mixed with barium chloride, and after standing for about forty-five minutes, the precipitate was first washed by decantation, and finally washed on the filter with boiling water. The washings did not get turbid on standing, and the ignited precipitates were all crimson-red.

Five experiments (*b*) were made. The filtrate, measuring about 100 c.c., was slightly warmed and mixed with ammonia, until it acquired a strong colour. After ten minutes the precipitate was washed on a filter with boiling water, until the filtrate gave not the slightest cloud with barium chloride.

Now, Obregia exactly carried out my plan : to choose a rather large funnel, which is so made that the filter exactly fits it, and the filtrate constantly fills its stem. The water must be really boiling, and squirted into the centre of the ferric hydrate so as to get a thin paste, and prevent formation of channels. Working in this way, both ferric and aluminic hydrates may be conveniently washed on a filter, without the troublesome decantation. Obregia found the washing to last from three-quarters to one and a-half hour, according to the quality of the filter-paper. The total volume of the filtrate seldom exceeded 250 c.c., so that there was generally no need for concentration before estimating the sulphuric acid.

The ferric precipitates were now each time dried and fused in a platinum crucible with perfectly pure soda. The fusion was carried out by placing the crucible in a perforated sheet of asbestos, and heating with a Bunsen burner.. This simple arrangement prevents the access of sulphury vapours, and renders unnecessary the use of a Berzelius lamp, as used in Jannasch's and Richards' experiments. The mass was now exhausted with water and tested as usual for a sulphate. Even after standing for twelve hours, not a trace of it could be detected. If, therefore, one only works according to my directions, the ferric oxide will be perfectly free from sulphur. The precipitation of the iron free liquid was done with barium chloride as usual. The barium sulphate, after being ignited, was always perfectly white.

Three experiments (*c*) were carried out. The ore was fused with ten times its weight of a mixture of two parts of soda and one part of nitre, in a platinum crucible placed in asbestos, with all the precautions recommended by Fresenius. It must, however, be observed, that this method requires more manipulating skill, and takes at least double the time of the aqua regia process, and that it also quickly spoils the crucibles. The following percentages of sulphur were obtained :—

	A Lunge's old method.	B Lunge's method, 1881.	C Fresenius's method.
	52·38	52·70	52·46
	52·38	52·14	52·49
	51·94	52·22	52·31
		52·26	
		52·39	
Average	52·23	52·40	52·42

From these results I now can, with the greatest of certainty, draw the following conclusions:—

1. My process of 1881, viz., the acid treatment of the pyrites, and removal of the iron by ammonia, before precipitating the sulphate, gives completely concordant results with the fusion process of Fresenius. Even when the first experiment (B) is rejected, the average still comes to 52·32 per cent., therefore differing only ·10 per cent. from Fresenius' figures, who, in his own laboratory, with his own process, got far greater differences, viz., from 43·74—44·04 per cent. so ·3 per cent.

2. By these proved facts, also by the direct testing of the ferric precipitates, it is shown that when working properly, no sulphuric acid is retained. In this view I am supported by Barbezat and Obregia.

3. The former opinion of Jannasch and Richards, that my process ought to be rejected from a purely scientific point of view, does not relate to my process of 1881. This process is, at least, quite as accurate as the one of Fresenius, but has, besides, the advantage of being performed in much less time (not without advantage to works-chemists), and many analyses can be carried on at once. It further saves the wear and tear of the platinum crucibles; and last, not least, it is applicable in cases when the ore contains heavy spar, which renders Fresenius' process unreliable. Therefore my process of 1881, on account of its easy execution and reliability, deserves the preference over others, in the estimation of sulphur in pyrites.

4. My older process, which does not provide for removal of the iron, gives, as I found in 1881, results which are too low, but only by ·17 or ·19 per cent. In 1889 I found it to be ·18 per cent. Jannasch and Richards got far greater differences, but this is most likely caused by their extraordinary system of prolonged and intense ignition, such as nobody would think of doing. The process may, therefore, be trusted to yield results quite accurate enough for technical purposes, but in more important cases of buying and selling large quantities, my new process is to be preferred.

ANALYSIS OF WATER FOR DOMESTIC PURPOSES.*

By Ferd. Fischer.†

There is scarcely a branch of analytical chemistry where exist so many different views as in water analysis. Since analyses of potable waters are mostly done by professional analysts, instead of medical men, the chemical work has certainly become more exact; but the conclusions drawn from it are still, in many cases, incorrect, and do not rest upon sound foundation. Since Koch's discovery of the cholera baccillus, many medical men have taken to the bacteriological method, which in itself is very praiseworthy, if not too exaggerated conclusions are at once drawn. Plagge and Proskauer are of opinion that the chemical composition of a water goes for nothing; all they require

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* Slightly abridged from the original.

† *Zeitschr. f. angew Chemie, No.* 18, 1889.

ESTIMATION OF EXTRACTIVE AND SPECIFIC GRAVITIES OF OFFICIAL TINCTURES.

By J. Spilsbury, F.C.S., *Pharm. Ch.*

The following summary of averages is based on estimations extending over a period of ten years. The tinctures from which the samples were taken having been manufactured, under my personal supervision, from fine specimens of crude drugs (not specially selected samples) and strictly in conformity with the formulæ of the British Pharmacopœia.

The evaporations were conducted in thin porcelain basins of about 2½ ins. in diameter, with the exception of some rectified tinctures which had a tendency to creep over the sides, in which cases glass beakers of about 2 oz. capacity were substituted. The respective residues were exposed on the water-bath to a temperature of 212° Fah. and weighed at the expiration of two hours:

	Extractive from 10 c.c. expressed in grammes.	Specific Gravity at 60° Fah.
Tincture of Aconite ..	·231	·855
„ Aloes ..	·588	·961
„ Arnica ..	·029	·842
„ Asafœtida	·643	·865
„ Orange ..	·335	·935
„ Orange from fresh fruit	·256	·894
„ Belladonna	·083	·926
„ Benzoin ..	1·526	·889
„ Buchu ..	·179	·939
„ Calumba ..	·094	·928
„ Indian Hemp	·430	·846
„ Capsicum ..	·080	·843
„ Cardamoms	·567	·944
„ Cascarilla ..	·100	·932
„ Catechu ..	1·068	·961
„ Chiretta ..	·118	·927
„ Cimicifuga	·208	·929
„ Cinchona ..	·470	·939
„ Cinchona (compound)	·439	·937
„ Cinnamon	·140	·928
„ Colchicum	·100	·926
„ Hemlock ..	·243	·942
„ Cubebs ..	·118	·846
„ Digitalis ..	·407	·939
„ Ergot ..	·200	·932
„ Galls ...	1 215	·978
„ Gelseminum	·118	·929
„ Gentian ..	·398	·939
„ Guaiacum	1·392	·939
„ Henbane ..	·299	·930
Tincture of Jaborandi ..	.232	·928
„ Jalap ..	·355	·940
„ Rhatany ..	·314	·937
„ Larch ..	·128	·846
„ Lobelia ..	·301	·939
„ Lobelia (Ethereal)	·105	·824
„ Hop ..	·267	·930
„ Myrrh ..	·366	·849
„ Nux Vomica	·122	·888
„ Opium (simple)	·416	·936
„ Pyrethrum	·153	·860
„ Quassia ..	·002	·921
„ Quinine ..	·532	·939
„ Rhubarb ..	·538	·955
„ Savin ..	·235	·933
„ Squill ..	·878	·953
„ Senega ..	·318	·931
„ Senna ..	·835	·893
„ Serpentary	·087	·911
„ Stramonium	·045	·939
„ Sumbul ..	·365	·931
„ Tolu ..	1·223	·880
„ Valerian ..	·208	·928
„ Valerian (Ammoniated) ..	·237	·907
„ Veratrium	·189	·847
„ Ginger (strong)	·197	·847

The inference deducted in obtaining the foregoing results indicates that tinctures manufactured under similar conditions should not vary from the average amount of extractive in the proportion of more than ·01 gramme for every ·5 gramme yielded, 10 c.c. of Tincture being used for the evaporation.

MILK ANALYSIS.

By Dr. Bennett F. Davenport.*

In the discussion of the relative merits of the different methods of milk analysis which was published in the June number of The Analyst, the many advantages of a simple modification of Wanklyn's method does not, I think, receive its due consideration. This modification, which I have adopted, I have already published in my Annual Report as Milk Inspector for the City of Boston, Mass., 1885, and as Analyst to the Massachusetts State Board of Health in their Annual Report for 1886, page 138. As being now the method generally followed by most of the official milk analysts in New England, a simple description of it may be of interest to others.

The 5 grms. of milk are weighed off in a large flat-bottom platinum capsule of full 2½ inches diameter on the bottom, and about 3 inches across the top. The ½-inch high side turns up from the bottom, not with a sharp angle, but slight rounding, this being about the curve with which the milk runs up the rim of the capsule drawn by capillarity. The milk in drying down does not thus form any thicker deposit at the angle of the side than elsewhere upon the about 5 square inches of bottom surface of the capsule. This relatively large amount of surface, one square inch to each grm. of sample taken, causes each inch to be covered with only a little over a single grain of dried milk solids. The deposit is therefore so very thin as to be readily exhausted of its fat in its subsequent treatment with boiling petroleum naphtha. When using such small dishes as were originally proposed by Wanklyn, the residue would be so thick, that is about three times, as would naturally render any such rapid method of extraction as I employ impossible, as the English analysts have learned. Each capsule has its serial number engraved upon it, and they are made to weigh a little over 25 grms. each, that the bottoms may be stiff enough to remain perfectly flat—a matter of very great importance. They are also made to differ from each other only in the second and third places of decimals, so that only those weights have to be changed in many weighings, which is a matter of no little saving of labour where many score of weighings are to be made each day. A table of their weights is kept within the case of the scales. These capsules, containing their samples, are placed upon a constant-level closed-top water-bath, of the peculiar construction described on page 269 of the *Journal of Analytical Chemistry*. This will hold twenty-five capsules at a time. Here, being surrounded by atmosphere not already nearly saturated with moisture, as would be the case if they were upon a water-bath with openings on its top, they quickly evaporate to apparent dryness. They are then transferred for their final drying to a constant weight to one of Weisnegg's large porcelain-lined air-baths, regulated to the constant temperature of 105° C. Here they are dried in about half an hour, when they are cooled in a desiccator and each weighed immediately upon being taken out, to guard against their rapid gain in weight from exposure to the air. Replaced upon the closed-top water-bath, the capsule is filled from a wash bottle with petroleum naphtha, of the quality of the benzine of the U. S. Pharmacopœia, revision of 1880. This, unlike ether, will dissolve out neither milk-sugar nor lactic acid from the dried milk-solid residue. Moreover, one gallon of it costs but as much as one pint of ether would, which is a matter of some little consequence to one like me, who has had to use about 100 gallons of it in milk analyse during the past five years.

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* *Journal of Analytical Chemistry.*

The naphtha in the capsule, after being allowed to boil down about one-half, i decanted off against a rod into a basin to guard against the remote possibility of some flakes of milk residue being poured off with it. Replaced upon the bath, the capsule is refilled with naphtha. This boiling up and decanting off is repeated three to four times, when after the last one the outside of the capsule is washed off with naphtha played upon it from the wash-bottle, to prevent any residue of fat being left there. The capsule, then finally replaced upon the bath to dry off the naphtha, is then cooled, and weighed as before, the butter being rather determined from the loss of weight i the solids than by the weight of the evaporated washings.

The ash is made by ignition of the capsule over a one-inch wide Bunsen lamp, which thus gives so wide a flame as not to require a heating to a high temperature at one point, and thus a possible loss by the volatilisation of the potassium chloride. The milk-sugar I determine by the use of a Soleil-Ventzke saccharimeter, in the same manner as has now been several times described in the journals by Drs. Wiley, Vieth, and others. The other ingredients which reduce copper in Fehling's solution, are not thus reckoned as sugar, by which the apparent amount of albuminoids present, which are obtained by difference, would be diminished.

The peculiarities of my modification is then the use of an evaporating capsule of such a very large relative area as will leave the residue thin enough to be readily exhausted by the boiling naphtha. Thus a diameter of 2½ inches in the capsule will do for 5 grms. of better than average quality of milk, with a surety that there will not be as much as one-tenth of 1 per cent. of fat left as a maximum error. And surely this is fine enough for all commercial work, and it is much to be doubted if much which affects to be more accurate by a decimal than this is really so. The method, as involving no transfer of substance, cannot well be made more simple for the determination of total solids, fat and ash. When the average life of such an evaporating capsule under reasonable usage is considered, it cannot well be cheapened, although the outlay for a kilo. of platinum for forty capsules like mine does, it is true, involve an original expense of nearly 400 dollars. The shape of my capsules makes possible completeness of extraction with the solvent used, and the construction of the closed water-bath hastens the process in no small degree.

ON SOME RESULTS OF ANALYSES OF OLIVE OIL FROM DIFFERENT SOURCES.

By Leonard Archbutt.*

The examination at various times during the past seven or eight years of samples taken from large bulks of olive oil intended for lubricating has afforded an opportunity o making a comparison of the oil from several different sources; but as, unfortunately only a fraction of the samples have been labelled with the port of shipment, the numbe available for purposes of comparison is not so large as it might have been.

The results are arranged in the following table, and the following conclusions ma be drawn from them:—

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* *Journal of the Society of Chemical Industry.*

Source of the Oils.	Total Number of Samples.	Number Examined for Adulteration as well as for Acidity.	Number found Genuine.	Per Cent. found Genuine.	Free Fatty Acid (=Oleic Acid).		
					Highest.	Lowest.	Average.
Seville	32	32	31	96·8	10·0	1·5	4·4
Malaga ..	35	35	27	77·1	25·1	2·0	6·7
Unknown ..	3	3	2	66·6	5·6	2·1	3·3
Spain (total)..	70	70	60	85·7	25·1	1·5	5·5
Gallipoli ..	19	18	17	94·4	16·9	·9	7·3
Gioja	12	8	8	100·0	25·2	4·1	10·9
Monopoli ..	3	3	3	100·0	8·6	4·1	6·3
Naples	2	1	1	100·0	9·4	9·2	9·3
Italy (total) ..	36	30	29	96·7	25·2	·9	8·5
Sicily (Messina)	28	22	9	40·9	16·6	·5	9·1
Greece (Zante)..	2	2	2	100·0	8·7	4·8	6·7
Candia	12	10	6	60·0	16·8	5·5	9·5
Levant	3	3	..	..	13·5	8·5	10·4

The Seville oils, both as regards their freedom from acidity, and the high percentage of genuine samples among them, prove to be superior to the famed Gallipoli oils. The acidity of some of the samples of Malaga oil was very high, but the average acidity was less than that of any of the Italian oils, except those from Monopoli, of which only three samples were examined. Among the Malaga oils there was a large percentage of adulterated samples. Taking the Spanish oils generally, although they appear from these results to be more liable than the Italian oils to be adulterated, yet, on the other hand, they are as a rule decidedly more neutral, which, as regards lubrication and burning, is a very important point.

Amongst the Italian oils those from Gioja are distinguished for their acidity. The small number of adulterated Italian oils is worthy of note.

My analyses of the oils from Sicily quite confirms their reputation for inferiority. They are very largely adulterated, and as a rule decidedly rancid.

A few single samples from sources not named in the table have also been examined. Thus, some oil from Mitylene proved to contain as much as 30 per cent. of free fatty acid. A sample labelled "Sfax Fine Olive Oil" gave the following results :—

Sp. gr. at 60° F.	·9169
Free fatty acid (=oleic)	13·5 per cent.
Rise of temperature with sulphuric acid of 97 per cent. strength	43·5° C.
Percentage of KOH required for saponification ..	19·38 per cent.
Melting point of fatty acids by capillary tube method	28° C. and 28·5° C.

The Elaidin required 310 minutes to solidify at 19° C., and was dark lemon yellow and soft after standing 24 hours at 19° C.

The oil showed no unusual tendency to dry when tested side by side with olive oil of known purity.

By Renard's process no Arachis oil was found.

By the Miliau-Stock test no cotton oil was found.

The melting point of the fatty acids is quite abnormally high, and so also is the specific gravity, considering the large percentage of free fatty acid in the sample. No conclusions can be drawn, however, from the results yielded by this single sample. Another African oil, from Saffi (Morocco) gave quite normal results.

DYNAMICAL THEORY OF ALBUMINOID AMMONIA.

By Robert B. Warder.[*]

The following investigation was suggested by committee work of the American Association for the Advancement of Science, on water analysis. It is an attempt to apply well established principles of mass action to certain questions involved in the determination of albuminoid ammonia. The following points have been kept in view:

1. Can some probable correction be made for the error incurred by stopping the distillation while ammonia is still coming off?
2. What modification of the original method seems rational?
3. What insight can be gained into the nature of the chemical process?

The experimental basis of the following paper will be found in Mallet's report on the determination of organic matter in potable water,[†] with the addition of some details kindly communicated by Dr. Charles Smart.

I. *Distillation of Free Ammonia.*

In the distillation of albuminoid ammonia, two stages must be distinguished: first, oxidation of the nitrogenous body by the alkaline solution of permanganate; and, second, the separation of ammonia thus formed by distillation. It will be most convenient to begin with the discussion of this second process, which is essentially physical. As in any other case of fractional distillation, water and ammonia both pass from the fluid to the gaseous or vapour condition, in accordance with their respective tensions and mass.

Let $x =$ volume of fluid in retort.
$y =$ weight of ammonia in retort.
$z =$ weight of ammonia in distillate.

Then $\frac{y}{x} =$ concentration of fluid in retort,

$\frac{dy}{dx} = \frac{-dz}{dx} =$ concentration of any small portion of distillate.

The coefficient of volatility may be defined as the ratio of the concentration in any small portion of distillate to that of the fluid in retort. Designating this coefficient as k,

$$\frac{dy}{dx} = k . \frac{y}{x}. \qquad (1)$$

---

[*] *American Chemical Journal.*

[†] Report of the National Board of Health for 1882.

In the ordinary distillation of ammonia, k is found to be approximately constant, nd it will be so regarded here. By integration,

$$\log_e y = k.\log_e x + \text{constant};$$

sing zero subscript to denote initial conditions,

$$\log_e y_0 = k.\log_e x_0 + \text{constant},$$

$$\therefore \quad \log_e \frac{y}{y_0} = k.\log_e \frac{x}{x_0},$$

or

$$\log \frac{y}{y_0} = k.\log \frac{x}{x_0}, \qquad (2)$$

very useful formula for determing the coefficient of volatility and tracing the progress f distillation. Curve A, in the figure, represents y as that function of x defined by quation (2).

Wanklyn,* who was the first to use and define the term coefficient of volatility, eports four experiments for its determination. One litre of dilute aqueous ammonia as distilled in each case until 50 cc. of distillate had been collected. As nearly one-alf the ammonia taken was distilled with the first 5 per cent. of water (the concen-tration in the retort being gradually reduced at the same time), Wanklyn estimated k qual to "about thirteen or fourteen." The experimental data are given below with ore precise values of k, as calculated by equation (2). In each case, $x_0 = 1{,}000$, $= 950$. The quantities of ammonia taken, and that found in the distillate, are xpressed in milligrams. under y_0 and z respectively; the difference (remaining in retort) nder y:

y_0.	z.	y.	k.
1000	480	520	12.74
1	·50	·50	13·50
·5	·235	·265	12·39
·2	·095	·105	12·55
		Mean	= 12·8

This value may be rather too low, as no account is taken of a possible loss from mperfect condensation, a loss averaging 7·2 per cent. in Smart's experiments.

The same author observes also † that in the distillation of one-half litre of water, he ammonia found in the first measure of 50 c.c. is three-fourth of the whole quantity f ammonia obtained. By equation (2), assuming that any loss affects each part in the me proportion,

$$\log \tfrac{1}{4} = k.\log \tfrac{9}{10},$$

$$\therefore \quad k = 13{\cdot}14.$$

Thirty experiments by Smart to determine the loss of ammonia by imperfect ndensation are also available for the estimation of k, the successive measures of stillate having been nesslerised separately.‡ A single distillation affords several lues, as each fraction determined may be compared with the whole quantity found in bsequent fractions. The values thus deduced vary from 10 to 16, with a few instances yond these extremes. The variations in the values of k do not show any well marked lation to the whole quantity of ammonia present, to the amount of loss, nor to the rate of boiling. The experimental work was done wholly for another purpose, and probably with just such care as the conscientious analyst would ordinarily give. We may then assume 13 as a mean value for k, remembering that considerable allowance must be made for unexplained variations.

---

* Phil. Mag. [4], 45, 132 (Feb. 1873).

† Wanklyn's *Water Analysis*, 6th and 7th editions, p. 41.

ost of these data appear in the Report of the National Board of Health for 1882, pages 312-314.

II. *Application of Formula to Estimation of Free Ammonia.*

In the analytical process it is usual to distill three or four fractions, each nearly equal to one tenth of the original volume. For 100 parts of free ammonia present, when $k = 13$, theory would lead us to expect, in four successive fractions, the increments named below under Δz, with loss of 0·13; vertical lines in the figure, a, a, a, represent these increments geometrically. The ratio of each increment to the next is stated under r. The mean values of the ratios obtained in the experiments to determine loss of ammonia are given under r':

No.	Δz.	r.	r'.
1	74·6	3·75	4·23
2	19·9	4·4	5·26
3	4·53	5·4	5·65
4	·84		

In the analyses of natural waters reported, considerable variation appears in the ratios of successive increments, but usually not more than might be expected from the observed variations in k. Analyses No. 18 and No. 54 are exceptional and happen to give identical results, the free ammonia being ·015, ·01, and ·01 mgm. in the three fractions nesslerised, with ratios 1.5 and 1. In such cases there is a strong probability that some more complex body is gradually decomposed by simple boiling with sodium carbonate. Small ratios between the successive fractions of "free ammonia" may lead to suspicion of urea contamination, though of course due caution should be used in drawing such conclusions. The analyst will at least find it instructive to enter the ratios in his note-book, together with the separate results of nesslerising; this will appear more plainly in the following sections. The slowness of the chemical action usually retards the distillation of albuminoid ammonia, giving rise to much smaller ratios between successive fractions than those named above.

III. *Action of Mass in Formation of Albuminoid Ammonia.*

We must now attempt to follow the chemical as well as the physical aspect of the process. In the former the reactions may be much involved, and various formulas may be required to represent the successive formation and destruction of different substances. Let us consider, as an ideal case, a single chemical reaction, involving one molecule each of three reacting bodies; the formation of ammonia taking place at each moment of distillation, in each cubic centimeter of the fluid, in proportion to the product of the quantities of the nitrogenous body, the potash, and the permanganate present therein.

Retaining the use of x, y, and z as explained in section I, let
u = weight of the nitrogenous body in the retort,
v = weight of potash in the retort,
w = weight of potassium permanganate in the retort,
t = time, in minutes, from the beginning of the reaction,
a = coefficient of speed.

ssume also a uniform rate of distillation, in which

$b =$ volume distilled per minute,

$bc =$ volume of fluid in retort at the beginning of the action; whence

$$x = bc - bt,$$

d

$$dx = -bdt.$$

hen $\frac{u}{x}$, $\frac{v}{x}$, and $\frac{w}{x}$ = weights of the three active substances in each cubic centimeter of uid in the retort,

$$\frac{-du}{dt} = b\,\frac{du}{dx} = \text{rate of change},$$

nd, by the law of mass action,

$$b\frac{du}{dx} = a.\frac{u}{x}\cdot\frac{v}{x}\cdot\frac{w}{x}\cdot x = avw\,.\,\frac{u}{x^2},$$

$$\therefore \frac{du}{u} = \frac{avw}{b}\cdot\frac{dx}{x^2}. \qquad (3)$$

The reagents are present in such large excess that v and w may be regarded as onstants (unless the permanganate suffers from an unusual amount of pollution), and y integration,

$$\log_e u = -\frac{avw}{b}\cdot\frac{1}{x} + \text{constant},$$

$$\log_e u_0 = -\frac{avw}{b}\cdot\frac{1}{x_0} + \text{constant},$$

$$\therefore \log_e \frac{u}{u_0} = -\frac{avw}{b}\cdot\frac{x_0 - x}{x_0 x},$$

hich may be written

$$-\log\frac{u}{u_0} = \frac{avw}{bx_0 \log_e 10}\cdot\frac{x_0 - x}{x} = A.\frac{x_0 - x}{x}. \qquad (4)$$

This equation represents any one reaction of the kind specified during the oncentration required in an ordinary estimation of albuminoid ammonia, but the athmetical expression must not be pressed too far. Thus, if we make $x = 0$, this not nly implies that the contents of the retort are evaporated to dryness, but also that the eagents have gained infinite concentration, and therefore act with resistless force, ffecting complete alteration of any remaining traces of the nitrogenous body. The orm of the curve varies greatly with the value of A. Two examples are given in the igure. Curve B represents this equation when $A = 1$; curve C when $A = \frac{1}{10}$. Since he quantity of ammonia present at any moment (both in retort and distillate) is proortional to the weight of the nitrogenous body transformed we may write

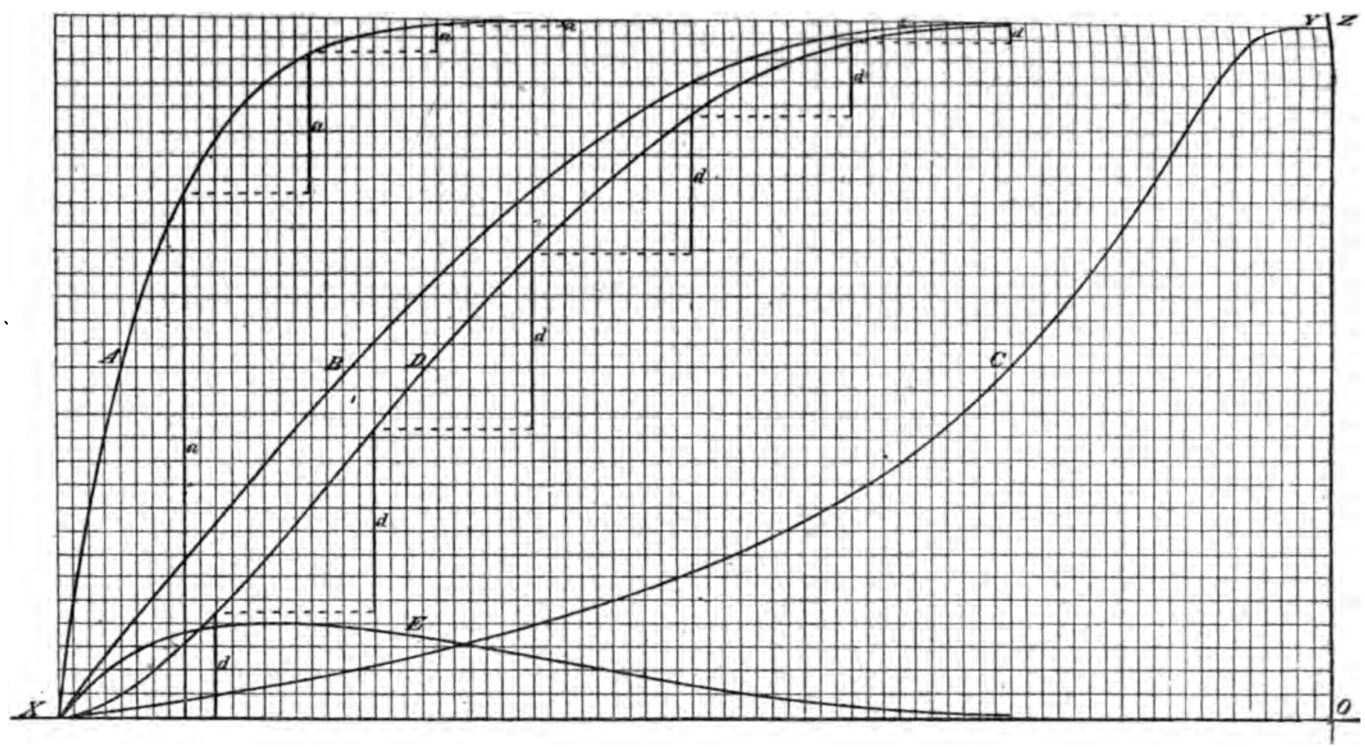
$$u_0 - u : u_0 :: (y+z) : (y+z)_\infty,$$

here subscript ∞ is used to denote the ultimate value theoretically possible; or, in nother form (since $y_\infty = 0$),

$$\frac{u}{u_0} = \frac{z_\infty - (y+z)}{z_\infty}. \qquad (5)$$

he curves B and C are so drawn that ordinates represent $(y+z)$ as a function of x.

It will be seen at once that when $A = 1$ the quantity of ammonia increases very pidly during the first part of the distillation, but when $A = 0{\cdot}1$ $(y+z)$ increases but owly until the fluid is considerably concentrated. Equation (4) suggests various modiations by which A may be altered at pleasure; for example, by increasing the quantity permanganate used (represented by w), or by distilling a less quantity (b) each inute. Thus Smart noted several instances of increased action by slower boiling.



EXPLANATION OF THE FIGURE.

A. Curve of distillation of free ammonia. *a, a, a*. Fractions, each $\frac{1}{10}$ the original volume. *B*. Formation of albuminoid ammonia, eq. (7); $A = 1$. *C*. Formation of albuminoid ammonia, eq. (7); $A = \frac{1}{10}$. D. Ammonia is distillate, from curve *B*. *d, d, d*. Fractions, each $\frac{1}{8}$ the original volume. *E*. Ammonia in retort, from curve *B*. Value of x, although positive, are measured towards the *left*, in order that time may be counted towards the *right*.

(*To be continued.*)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

THE SEPARATION OF COPPER FROM ANTIMONY. FINKENER. *Mitth. Königl. tech. rsuch*, 1889.—The separation is effected in two stages: first by the precipitation of e bulk of the copper, as cuprous iodide in an acid solution, and then by the removal the remainder as sulphide in an ammoniacal solution. The author has established e following, which serve as a basis for the method:—(1.) Cuprous iodide carries down timony even in the presence of tartaric acid from a nitric acid solution of the two etals, but does not do so if the antimony be first converted into a double salt of timony pentafluoride and an alkaline fluoride by the addition of the latter. (2.) Free dine in the presence of an alkaline fluoride converts antimony trifluoride into the entafluoride. (3.) Sulphurous acid reduces antimony pentafluoride slowly in the resence of potassium iodide; the addition of hydrochloric acid hastens the reaction, ut the presence of potassium fluoride almost completely inhibits it.

From this the following method is derived:—If the solution to be treated contain he antimony as a pentad compound, all that is necessary is to add an alkaline fluoride, hen potassium iodide and sulphurous acid; heat gently, filter off the bulk of the opper as cuprous iodide, and wash with boiling water, made acid with sulphuric acid. n the event of the antimony being in the triad state the solution may be first treated ith chlorine or bromine water, or oxidation may even be effected by the iodine iberated by the action of the cupric salt also present on the potassium iodide, provided ime be allowed for it before the addition of sulphurous acid. The filtrate containing ll the antimony and a little copper is oxidised by bromine water, and the metals pre-ipitated together by sulphuretted hydrogen, the mixed sulphides dissolved in hydro-hloric acid with the addition of potassium chlorate, ammonium tartrate added, and mmonia in excess; the resulting solution is heated on a water-bath and sulphuretted ydrogen added, until no further precipitation occurs. By thus minimising the quantity f ammonium sulphide present, re-solution of cupric sulphide is avoided. The filtrate ontaining only the antimony is acidulated with sulphuric acid and sulphuretted ydrogen passed through; the antimony is determined in the sulphide thus precipitated y heating it, together with its filter, with hydrochloric acid, and determining the ulphuretted hydrogen evolved by standard iodine solution. The author has established hat antimony sulphide, precipitated in the presence of sufficient ammonium tartrate to onvert all chlorine present into ammonium chloride, is of normal composition, and ields the theoretical quantity of sulphuretted hydrogen when heated with hydro-hloric acid. The analytical results show the method to be reliable, if potassium or odium fluoride in the proportion of about 1 grm. to 0·275 grm. of antimony be used, nd the solution in which the first stage of the separation is effected be about 200 c.c.

W. H. D.

ABNORMAL APPEARANCE OF CHOLERESTIN. J. B. NAGELVOORT. *Nederl. Eydschr. v. harmacie, etc. October*, 1889.—A sample of cod liver oil submitted to the author was sted in the usual way for phytosterin, and, indeed, the well-known needle-shaped ystals were obtained, apparently showing the presence of a vegetable fat. On applying e chemical tests, it was, however, proved to be cholerestin. On repeating the process, the author succeeded in getting the proper crystals, but he fears their shape may be influenced by temperature, concentration of the liquid, and rate of evaporation, and, therefore, the mere appearance counts for nothing. The following reactions serve to recognise cholerestin: Sulphuric acid gives a reddish-brown, turning to dirty green on adding a drop of water. A mixture of equal parts of chloroform and sulphuric acid gives a violet colour, changing to red and immediately discharged by a drop of water.

L. DE K.

ON THE ANALYSIS OF OLIVE OIL. RIOUL BRULLE (*Rev. Internal. des Falsification,* Oct. 15, 1889).—For the testing of olive oil, the author employs argentic nitrate in the presence of fuming nitric acid. The argentic nitrate decomposes violently, and produces metallic silver, with a coloration depending exactly on the nature of the oil employed. If we mix 10 c.c. of an oil with 0·5 c.c. of fuming nitric acid in a porcelain capsule, heat, and shake the mixture violently until a foam has been produced, we obtain different colours, according to the oils employed. We, however, take no notice of these, but go on with the process by adding 5 c.c. of a 25 per cent. solution of argentic nitrate in alcohol of 90°. Still applying heat, a moment arrives, at about 115° C, when the argentic nitrate decomposes, and deposits metallic silver. Having reached this point and continued to heat until the first flecks have just disappeared, we observe, on the one hand, the coloration of the slight oily stratum, which is easily seen by slightly inclining the capsule; and on the other hand, the metallic flecks glistening on the surface of the liquid. On saponifying the oils, and treating them by the same method, the colorations obtained are all different, as may be seen by the following table:—

Oils.	Colorations obtained.			
	In the natural state.		Saponified.	
	Oily strata.	Flecks.	Oily strata.	Flecks.
Olive	Olive green	Green	Orange	Brownish green
Cotton	Green	Terra vert	Raw sienna	Cobalt violet
Sesame	Chrome green	China blue	Gold yellow	” ”
Ground-nut	Sap green	Emerald green	Persian lake	Bright violet
Poppy-seed	Olive green	Pale greenish blue	Gold ochre	Blue
Gold-of-pleasure	Persian lake	Bright blue	Deep chrome	”
Linseed	Dragon's blood	Emerald green	Black	Green
Colza	Persian lake	Brownish green	Burnt carmine*	Ultramarine blue

* After becoming cold, it forms a crystallization of blue-coloured needles on the surface.

The designations of the colours are those used in water-colour painting. In comparing the tint of any mixture of oils treated by this method with those given in the above table, a chemist will, after a little experience, be readily able to prove the presence of a seed oil in olive oil, as well as to detect adulterations as low as 5 per cent., and to name the foreign oil present in the sample.

M. S. A. M.

The Quantitative Estimation of Galactose. By E. Steiger (*Zeits. f. Anal. em.* 28, 444).—The author has prepared a table to calculate the quantity of galactose om the quantity of copper reduced; 60 c.c. of Fehling's solution diluted with an equal lk of water is brought to boiling; 25 c.c. of the galactose solution, previously warmed, added, and the mixture heated 3-4 minutes. The filtration was done by Soxhlet's bestos Filter (see Fresenius' "Quantitative Analysis," 6th edition, p. 596), and the pper oxide reduced in a current of hydrogen. The time—3-4 minutes—must be hered to, Allibon (*Journ. f. Prakt. Chem.* 22, 72) having shown that by long heating alf an hour) a further precipitation of cuprous oxide occurs. The author's experients show that from 3 to 7 minutes' heating, the results are the same.

The following table gives the mean of his results, obtained from two different mples of galactose, four estimations being usually made for each dilution:—

Galactose.	Copper.
250 m.g.	434·5 m.g.
237·5	411·8
225·0	393·6
212·5	375·0
200·0	354·2
187·5	335·0
175·0	316·4
162·5	297·6
150·0	277·5
137·5	254·0
125·0	232·7
112·5	211·1
100·0	188·7
87·5	165·4
75·0	142·4
62·5	120·2
50·0	94·8
37·5	73·1
25·0	49·9

H. D. R.

REVIEWS.

Coloured Analytical Tables Showing the Behaviour of the More Common Metals and Acids to the Ordinary Reagents, with Special Reference to the colour of the Various Oxides, Salts, Precipitates, Flames, Borax-beads, and Blowpipe Reactions. A Class-book for Students in Hospitals, Colleges, and Schools. By H. Wilson Hake, Ph.D., F.I.C., F.C.S. London: G. Philip & Son, Fleet Street.

This book, with so voluminous a title, and a preface nearly filling two pages, is simply a set of the usual sort of imperfect analytical tables served up to the budding aspirants for ordinary medical primary examinations, and sufficiently full to get them through heir arduous analytical labours in the (so-called) class of practical chemistry. It, however, differs from all other books with which we have met (except Dr. Simon's "Manual f Chemistry," published in 1885 and reviewed at the time in our columns), inasmuch s that, attached to each test there is a picture of a test tube showing a hand-painted epresentation of what the colour of the precipitate should be (supposing, of course, that he student works properly); and there are also similarly coloured representations of lowpipe beads, and charcoals, and of bunsen flames. What real advantage there will be to the student other than enabling him to skulk his practical chemistry class even more than he now too often does, and grind up at home, it is difficult to see. But that it will be useful to the unfortunate demonstrator who is expected to teach practical chemistry to perhaps thirty men all at once, is quite evident. This is the real use of such illustrated books; and Dr. Hake lets the cat out of the bag in his preface, when he says: "The book, I believe, should prove of service in schools, or in large classes where individual attention from the teacher becomes difficult, or almost impracticable; and medical students who, under the present regulations, have a good deal of simple analysis to learn in a comparatively short space of time, will possibly find their labours lightened by its use." There is no doubt that all those hospital and school teachers who like to take the least possible trouble over their work will find it well worth their while to quietly introduce Dr. Hake's illustrated tables to the notice of the class. Like many such books, it is here and there a little slipshod, as witness the paragraph on metals, in which a student, having been told that he must not use HCl, but nitric acid, to dissolve Pb, Hg, and Cu, is yet left in doubt as to whether he might not use hydrochloric acid for Ag. Surely it would have been just as easy to put the Ag in the nitric acid set and be done with it. So far as the colouring goes, it is really very well done indeed, and quite amusingly natural in many cases, and as we said before, the book is a real boon to the overworked or lazy teacher, and to the "wagging" student.

LAW NOTES.

CONDENSED SKIM MILK.—An oilman named White, carrying on business in Camberwell-road, was summoned at Lambeth Police Court, for selling condensed milk not of the substance and quality represented, according to the terms of the Adulteration Act.—Mr. Biron, barrister, instructed by Mr. Marsden, vestry clerk of Camberwell, appeared to prosecute; the defendant was represented by Mr. Besley.—George Dewey, one of the inspectors of the Camberwell Vestry, stated that he went to the defendant's shop and asked for three tins of condensed milk. White asked him what brand he would have, and he replied the "Standard" brand. There were several other brands there. He paid 9d. for the three tins. Witness now produced the certificate of Dr. Bernays, the analyst for Camberwell, which showed that 90 per cent. of the cream had been abstracted.—Dr. Bernays in his evidence stated that the certificate produced was given by him. The label upon the tins sold was as follows: "The Standard Brand is specially prepared from the richest cow's milk from which a portion of the cream has been removed, and nothing added but cane-sugar. It is better and cheaper than fresh milk for ordinary purposes. Five parts of water to one of milk, and for infants 8 to 15 parts water, according to age." There had been nothing added to the milk but cane-sugar.—By Mr. Biron: Such a preparation so diluted would not be fit food for infants.—Mr. Besley said the "Standard" brand was a good milk, and then proceeded to quote several decisions with regard to such cases, and contended that the defendant was not liable.—The defendant was called and stated that he sold the milk in question and other brands at 3d., 4d., 4½d., and 5½d. He declared that he told the inspector it was partly skimmed, and was sure he called the attention of the officer to the labels on the tins, which showed it was skimmed milk.—Some legal arguments followed, and Mr. Biron (the magistrate) said it no doubt was an important and difficult case. Under all the circumstances, however, he felt bound to convict, although at the same time he was fully satisfied the defendant was without the slightest blame or had any intention to defraud. He should, therefore, only be too willing to grant a case for the various points to be argued in a superior Court. He considered the sale was one to the prejudice of the purchaser under the Act, and he therefore ordered defendant to pay a fine of 20s. and costs.—Notice of appeal was given.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

SIR,—In your current issue you are correct in ascribing to me the first mention in your columns of the phenolsulphonic test for nitrates and the napthylamine test for nitrites in water (Vol. x., p. 190; Vol. xii., pp. 50, 152). It will be seen by reference to my papers that the extract from Dr. Leffmann's book approaches very nearly to a copy of my words. The same weights are given for preparing the reagents, and the same precautions directed to be observed. The only practical differences I have noticed are, the introduction of a glass rod with which to stir the mixture, and an error in the chemical formula.

I am, etc.,
A. PERCY SMITH.

Phœnix Mills, Dartford.
October 3rd, 1889.

THE ANALYST.

DECEMBER, 1889.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A MEETING of the Society was held at Burlington House on Wednesday, the 13th ult., the President, Mr. Adams, in the chair.

The minutes of the London meeting in June and the Liverpool meeting in August were read and confirmed.

Mr. W. H. Collins, Analytical Chemist, of Bolton-le-Moors, was proposed as a member.

Mr. ADAMS read a paper "On a New Form of Air-bath," a model of which he exhibited and explained.

Mr. ALLEN read a paper "On Possible Future Extensions of the Duties of Public Analysts."

A prolonged discussion ensued on this, and ultimately, on the proposal of Dr. Muter, seconded by Dr. Johnstone, it was resolved "that Mr. Allen's paper be printed and circulated with the agenda for the next meeting, and that the discussion be adjourned until that meeting."

A paper by Dr. H. Leffmann and W. Beam, "On Some Applications of Centrifugal Action to Laboratory Work," was also read.

The next meeting of the Society will be held on Wednesday, the 11th inst., at 8 o'clock.

A NEW FORM OF AIR-BATH.

By M. A. Adams, F.R.C.S., F.I.C.

(*Read at Meeting, November,* 1889.)

Bearing in mind the universal and indispensable utility of the air-bath to the chemica analyst, one might expect to find much variety and perfection in the design and construction of such an important piece of apparatus. Strange to say, this is not so. I very much doubt if there is to be found any piece of chemical apparatus in the chemist' laboratory that has received less attention, or stands more in need of it.

This fact was forced upon my attention by the great difficulty found in bringing certain hygroscopic substances to a constant weight, and I soon discovered that the attempt was hopeless with the ordinary instrument, the reasons for which were not far to seek. In the first place, in the ordinary bath it is impossible to maintain a uniform temperature throughout the whole of the drying chamber, for, even with the help of a thermostat, though it may be regulated with accuracy for some one special portion, other parts will in all probability be found to be several, indeed many, degrees hotter or colder according to the circumstance, so that to dry a substance, say at 100° C., the bath, though regulated to stand at this point for the spot where the thermometer is placed, is no assurance that the thing to be dried is exposed to the temperature desired, and if the object is of any considerable bulk, the probability is that one portion of it may extend into a region that is much below a hundred, and another into a place that is much above a hundred. Naturally the greatest heat is found near the floor, where the thermometer is never placed, and the least heat at the sides and in the corners, where radiation and stagnation mostly takes place; but more especially it is cold in the line of draughts that proceed from the chinks of the door and through the primitive contrivance that is usually provided for ventilation. In these parts the temperature may be very little above that of the external atmosphere. No wonder then that the complete drying of delicate hygroscopic organic substances is found to be so difficult, for before the colder part can be made to give up the last trace of moisture that clings so tenaciously to it, the hotter is over dried and stands the risk of being charred.

In a case like this there is nothing to be got by turning and twisting the thing round so as to expose alternately its various parts to the greater heat. In imagination one can see what occurs; by a process of distillation and condensation the same kind of transference of moisture from the hotter to the colder parts takes place as is seen to occur, for instance, when the attempt is made to dry a damp flask under similar circumstances.

But even this picture by no means exhibits the whole of the perplexing difficulties of tracing and controlling the air currents in an ordinary air-bath. They are so erratic, so fortuitous, so delusive, and subject to such uncontrollable shiftings, that, practically speaking, it is a question whether thorough drying in such a case is possible without destruction of the substance. At all events the chances in favour of it are so slender as not to be relied upon.

Reflecting upon these difficulties, the theory of a perfect method for air-drying suggested two things as indispensable.

1. A constant current of pure dry air brought to the desired degree of temperature *before* admission into the drying chamber.

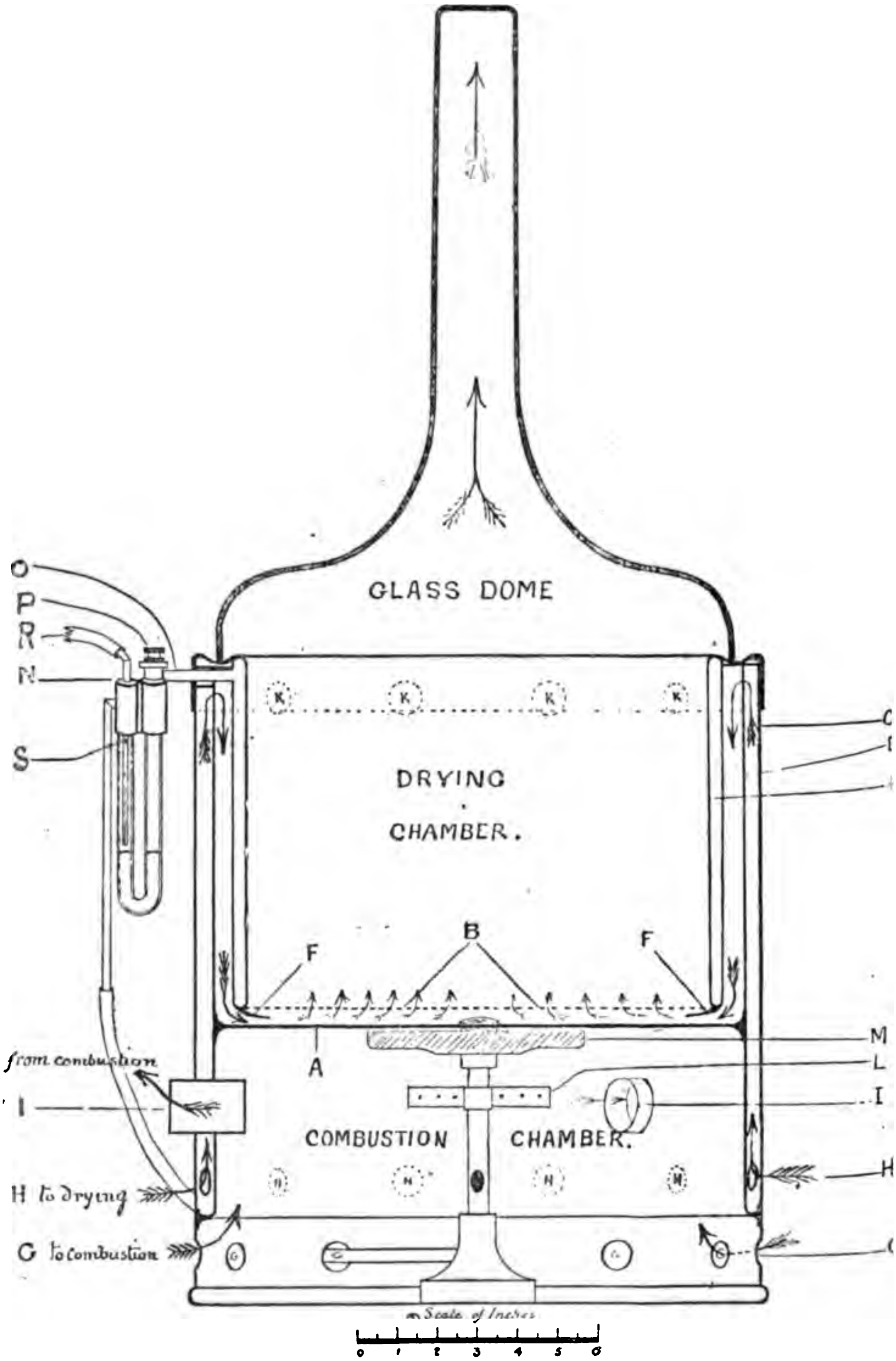
2. A regulated source of heat.

Both these conditions to be under perfect control.

I place current first as being the more important item, and the one to which hitherto very little attention appears to have been paid so far, at all events, as regards the temperature of the current when admitted into the drying chamber. As for the source of heat, this has been brought fairly under control by previous experimenters by the use of one or other of the several thermostats, though I was unable to discover one quite suited to the purpose.

To meet these requirements I have designed the instrument which I now have the honour of bringing under your notice, and which with the assistance of the diagram will be easily understood.

You will observe it consists essentially of a double jacketted cylinder with air passages, so contrived as to compel the air used for the drying to circulate between the jackets before its final admission into the drying chamber, in such a manner as to ensure that its temperature shall be raised to the required degree before it is allowed to come in contact with the thing to be dried. Moreover, as a secondary, but still very important consideration, the inlet of the air-supply is so placed that the air used for drying is kept as distinct as possible from all contact or admixture with the products of combustion, such products naturally being loaded with watery vapour and CO_2. The pure air thus heated before entering the drying chamber is evenly diffused over the whole area of the bottom of the bath, between it and the perforated false bottom which forms the floor of the drying chamber, the air then ascends bodily as a solid cylinder, and escapes by the tall chimney of the domed glass cover. In this way vigorous circulation of dry hot air is constantly maintained, which effects a rapid and uniform drying, such as no ordinary air-bath can accomplish. We will now turn to a consideration of the source of heat and its regulation. This consists of a Bunsen air-burner placed in the cavity under the bottom, but the heat is first received upon a solid disc of metal, separated by a sufficient space from the bottom to prevent the heat of the flame being transmitted direct to the bottom of the bath, the object being to avoid any localisation of the heat. Moreover, the mass of the metal disc, besides acting as a distributor, also serves as a reservoir of heat and assists in maintaining the equality of temperature, but this equality is chiefly provided for by an entirely new form of thermostat. It is to be observed that within the two jackets already spoken of is placed an annular copper vessel, which forms the boundary wall of the drying chamber. This is a cylinder composed of two thicknesses of thin sheet copper, enclosing an air space of 5 m.m. wide, 98 c.m. circuit, and 22·75 c.m. in height. It is securely closed top and bottom, and has a capacity of 1100 c.c. This constitutes the heat regulating chamber or thermostat, the cavity of which is connected up to a U tube, having mercury in the bend. Gas is admitted on the other side of the U, and by means of an arrangement, such as is usual in a gas thermostat, depression of the mercury in one limb cuts off the main gas supply, which can then reach the burner by a small bye pass only. By means of a screw at the top of the other limb of the U, air can be admitted into or allowed to escape from the regulator. With the rise or fall of the temperature, and the consequent expansion or contraction of the air contained in the regulator, pressure is exerted or withdrawn from the surface of the mercury, which is thereby forced down the one limb and up the other.


O
P
R
N
S
GLASS DOME
K
K
K
K
DRYING CHAMBER.
B
F
F
A
M
L
I
from combustion
I
COMBUSTION CHAMBER.
H to drying
G to combustion
H
C
Scale of Inches
0 1 2 3 4 5 6

A. Diaphragm completely separating the drying from the combustion chamber. B. Perforated bottom. C. Outer jacket. D. Inner jacket. E. Copper regulating chamber or thermostat. Baffle plate. G. Apertures in jacket C giving admission to air for combustion. H. Apertures for passage of air between the jackets for drying. I. One of three apertures for escape of products combustion. K. Apertures in inner jacket D for passage of drying air. L. Burner. M. Thick al plate for receiving heat of the flame. N. Mercury U tube. O. And its connection with per regulator. P. Screw whereby the degree of heat is regulated. R. Gas supply. S. Tube h bye-pass.

I have already mentioned the capacity of this copper regulator is 1100 c.c., the fficient of expansion for one degree Centigrade being ·00367, the alteration of volume a single degree of temperature at boiling point will be about three cubic centimeters 95). It is therefore plain we have here a means of regulation of the temperature of traordinary sensitiveness, and accordingly we find we can command what practically ounts to a fixed temperature at any desired degree, and seeing that the copper gulator entirely surrounds the drying chamber and that the whole of the air employed the drying process must of necessity sweep both its surfaces, exterior and interior, ounting to nearly half a (·4459) square metre in extent, it follows that no local rrents can interfere with the accuracy of its working. You will agree, I am sure, at this a grand point.

A Page's regulator, or any similar instrument, may be all very well in a still mosphere, but where a current is concerned it is not unlikely to be at fault and rown out of working from one cause or another, purely local, such for instance as ing shadowed by an object in process of drying, or being placed where there is either undue amount of current, or too little, or in an eddy. Our arrangement has a rther advantage of occupying no space within the drying chamber. Having said this uch respecting the principles involved in the design and the mode of construction, let now pass to a consideration of its performances; but before doing this it will be as ell to relate some particulars concerning difficulties encountered in connection with the gulator. When first set in action there was no getting a fixed degree of heat; the ermometer kept steadily mounting, degree by degree, without apparent cause. aturally we looked for some escape of air from the chamber of the regulator, but the sest inspection failed to reveal any point at which escape could take place, and it was ly by immersing the copper regulator in water and blowing through the tube attached at enabled us to discover several tiny leaks in the solder. After these were made od and the test repeated, the thermometer still recorded a constantly increasing temperare. Again and again we went through the process of searching for leaks, but all in in. Fixity of temperature seemed impossible, when at last I observed some condention of moisture within the U tube on the regulator side of the mercury, This at ce gave the clue to the cause of the rise of temperature. Each time of immersing e regulator in the water, when search was being made for leaks, a small amount of oisture must have gained access into the interior, and this, as the temperature of the th was raised to boiling point, became converted into steam and mingled with the ntained air. So long as this moisture remained at the high temperature of the terior of the regulator, it exerted the vapour tension due to that temperature, but tle by little a certain portion found its way into the U tube out of reach of the heat, d thereupon deposited its moisture by condensation on the sides of the tube, producing of course a partial vacuum in the tube and thereby drawing in a fresh supply of hot moist air and steam, so that at last quite a considerable amount collected in the U tube.

Now, seeing that 1 c.c., of water at 15·5 C. will produce 1696 c.c. of steam at 100° at ordinary barometric pressure, there is no need to dwell further upon the cause of our difficulty, or the necessity for keeping the interior of the copper regulator quite dry. I hope to be pardoned this digression. My excuse is that addressing practical men like yourselves one is bound to put those who may desire to follow in a similar investigation on their guard against needless loss of time, trouble, and temper.

Now, with respect to the performance of this instrument as an air-drying bath, I have directed my experiments to the demonstration of three things:—

I. To show the existence of, and determine the amount of, current passing through the bath.

The passage of the current is roughly but abundantly demonstrated by holding a flame opposite any one of the twelve air inlets; you will observe how the air rushes in. Again, at the outlet the current is manifested by this mica whirlygig arrangement, which you observe sails round famously by the impact of the current.

I have attempted to measure the amount of air that passes through the instrument by means of an anemometer, and find that it travels along a chimney whose sectional area = 5·4119 inches at the rate of 204 feet per minute, from which I calculate that no less than 7·6875 cubic feet of air pass through the apparatus per minute.

II. The next point of importance was to ascertain that this current was evenly distributed throughout the whole sectional area of the drying chamber.

This equal distribution you will observe was contrived at by making the instrument circular and admitting the air at points placed at equal intervals all round, and by surrounding the lower part of the inner jacket with a curved flange projecting inwards, the object of which is to direct the current horizontally between the true and the false bottom, and so prevent its premature passage through the perforations of the false bottom before having had time to take up heat from the bottom plate, and by thorough mingling and mixing, preventing local inequalities of temperature.

That these designs work well can be demonstrated by the smoke of smouldering brown paper, which shows that the current spreads itself over the whole area; there is no creeping up the sides or centre, it seems to pervade equally the whole space.

III. The final point that we have thought it important to enquire into relates to the vertical distribution of the heat.

At one time I was strongly tempted to head this communication with the title of "A Perfect Air-bath," and should have ventured to do so but for the practical impossibility of obtaining a perfectly equal temperature from top to bottom. For a certain very considerable range it is sufficiently so for all practical purposes, and far more so than it is, or can be, in any ordinary bath. For the convenience, if not the necessity, of the case, the source of heat is applied to the bottom, and you will remember we have interposed a large mass of metal between the flame and the bottom for the purpose of moderating, storing, and distributing the heat; but, nevertheless, all parts in metallic connection therewith get hot by conduction more in proportion as they are near to the source of heat. They in turn become radiators, and any object placed within near range of their radiation before the air current has had time to take up and distribute the same, gets more than its share of heat. Our experiments show that the useful range is anywhere above 3 ins. of the bottom. Below this undoubtedly the temperature increases rapidly, and more so the closer the bottom is approached. Above 3 ins., and for the whole of the rest of the drying-chamber the extent of the variation between any two parts does not amount to more than from 1° Centigrade.

In these several ways we have endeavoured to meet the requirements of a theoretically perfect bath.

We have contrived an instrument that provides a vigorous current of heated air of definite temperature under perfect control of a self-acting source of heat, and out of reach of contamination by the results of combustion.

Taken together, these results are, I submit, satisfactory, and show the instrument to be a substantial improvement on the ordinary air-bath, of sufficient value, I trust, to deserve your attention.

The following table exhibits the temperature as shown by two thermometers, one being fixed at 8 ins. from the bottom, which uniformly registered 100 degs. Cent., and the other at various distances as follows:—

Distance in inches.	Temperature degs. Cent.	Distance in inches.	Temperature degs. Cent.
10½	100	5½	100
10	100	4½	100
9½	100	4	100¼
9	100	3½	101
8½	100	3	102
8	100	2½	104
7½	100	2	106
7	100	1½	110
6½	100	1	115
6	100	½	124
5	100		

The metal portion of the apparatus was made by James Clinch, of 30, King Street, Maidstone, and the glass dome by Messrs. Powell, Whitefriars Glass Works, E.C.

Discussion.

Mr. Allen (who had temporarily taken the chair) said they would be very glad to inspect more closely this very ingenious and next door to perfect air-bath which Mr. Adams had described. They were indebted to Mr. Adams for several ingenious and practical devices. They would always associate his name with the paper-coil method of determining fat in milk, and they knew how thoroughly satisfied they were with it.

He would like to know how long it took before the desired temperature was obtained, because it was so extremely sensitive that the regulation of it might, he hought, prove troublesome in practice. He thought Mr. Adams might make the temperature more equable than it was by having an asbestos card-board diaphragm between he source of heat and the copper cylinder.

The contrivance by which Mr. Adams got rid of the heat afterwards was very teresting, because he got a good flow of heat over the bottom. He should have ought it was not necessary to have so large a copper cylinder to go over the interior of e bath, as it had a tremendous surface.

They were also indebted to Mr. Stansell for rigging up the apparatus there, and he should like to know where to get such glass hoods ready made, and he asked if it would not be convenient to have a regulator of the current, by means of a kind of damper at the top.

Dr. Muter said he thought this would turn out a very useful improvement in laboratory apparatus. It was practically an ingenious circular modification of the principle of Griffin's air-bath, which he had used for many years, and which was doubtless at present the best bath in the market; the air being taken in at the top, entering the inner chamber through holes at the bottom, and finally escaping by the chimney. He had much improved the working of his bath by placing a sheet of asbestos millboard over the bottom plate against which the burner impinged, and he thought, if the President would try such an addition, he would find that the unduly high temperature on the gauge bottom would no longer exist. If to this were added a second internal gauge, say two inches above the bottom one, for articles to rest upon, he should fancy that in the President's bath chemists would find the nearest approach to perfection yet met with in such apparatus.

Dr. Vieth said that Griffin's bath, so far as he knew, was at the present time the best in the market. He had used it for nine years with very little trouble and good results. He would like to ask Mr. Adams whether it would not be advantageous to place the false bottom three inches above the present one; that is to say, at a height where the temperature, according to Mr. Adams' observations, was, so to speak, normal.

Mr. Adams, in reply, said that it was by no means an expensive or complicated affair, and they must remember that it was not only a bath, but a thermostat as well, and perhaps it was the copper chamber which constituted the thermostat that made it appear somewhat complicated. All the rest was plain enough. In every instrument that he had seen, the means for escape of the products of combustion of the gas used for heating, was placed below the inlet for the air used for drying. This was a very serious fault. They could see (as he showed) what an enormous amount of watery vapour came out by the outlets provided for the escape of these products, and in the ordinary bath this went straightway into the drying-chamber, to say nothing of carbonic acid. Replying to Mr. Dyer as to whether there was any reason for having the dome made of glass, Mr. Adams said it was really a mournful thing to him that through the model having been broken by the railway company he was unable to show it in its entirety; the glass not only looked well, but everything that was going on could be seen, and the drying process watched without disturbance; but, of course, the glass dome could be replaced by a metal one.

As regarded regulation of temperature, this was as simple as possible. It could be set at any temperature wished for, from that of the room up to any degree that could be required, in the course of a few minutes. He had only to undo the screw (P) and allow a little of the air contained in the copper chamber to escape, and when the desired temperature was reached, screw it down again, and this, by preventing further escape, fixed the temperature at that point. On the other hand, if he wanted to lower the temperature, he would turn the gas out and allow air to enter the copper chamber until the temperature stood at the desired point. A thermometer hung from the chimney, and the temperature could be seen at any moment. The regulation could be accomplished not only exactly but immediately, and, moreover, the temperature was absolutely fixed. It might be set going on January 1st and go on to December 31st, and it would not vary. It might do some good to put some asbestos on the upper surface of the diaphragm that divides the drying and combustion chambers.

(*Conclusion of the Society's Proceedings*)

ON THE DETERMINATION OF CHLORINE IN WATER.

By Allen Hazen.*

ILORINE is almost always determined in water by titration with silver nitrate solution, ing potassium chromate as indicator. It has been my practice to titrate fifty cubic ntemeters of water or sewage in a porcelain dish with a silver solution of such strength at one cubic centimeter corresponds to half a milligram. of chlorine so that each bic centimeter used represents one part of chlorine in 100,000. The results mmonly obtained by this method are somewhat too high, owing to the fact that the d colour of silver chromate does not become perceptible until a certain excess of silver s been added, and this excess, as will be shown, is much too large to be neglected when ose work is desired.

I have made a large number of experiments to ascertain the amount of excess of lver solution required to give an end-point under various conditions of titration, and specially to find the influence of the volume of the liquid titrated, the amount of hromate used, and the amount of precipitated silver chloride present. I have found hat under constant conditions the required excess is constant, and that by making the roper corrections very accurate results can be obtained, even in widely differing conlitions of titration.

The general method by which these experiments have been carried out is as follows: ilver and chlorine solutions were made by dissolving the theoretical quantities of pure ilver nitrate and sodium chloride in distilled water. A solution of potassium chromate, ree from chlorine, was used which contained 0·12 gram. per cubic centimeter. Measured uantities of chlorine and chromate solutions were put into a white porcelain dish with istilled water, and titrated with the silver solution. The amount of silver solution required was in every case more than an equivalent of the salt used. The excess is the mount of silver solution required to reproduce an end-point under the conditions of the xperiment. In all cases I have not gone beyond an end-point which was as faint as ould be seen with certainty after some seconds, there being always a second dish of hromate, for comparison, by the side of that in which the titration is made. The olution should not be so concentrated as to make the precipitated silver chloride coagulate, s in that case the required excess is somewhat greater than otherwise.

The amount of chromate used has a considerable influence on the result, especially a large volume. In general, it may be said that the greater the amount of chromate sed, the smaller will be the required excess of silver; but when the quantity has ached a certain point, the advantage gained by any further increase is more than ercome by the increase in colour, which obscures the exact end-point. I have found 0010 gram. potassium chromate per cubic centimeter of liquid titrated (a much larger nount than is generally recommended) to be a suitable quantity. This is enough to ve most of the advantage due to increased quantity, while the end-point remains very arp. In practice I use 0·06 gram. of potassium chromate in all cases, even when the lume of liquid titrated is small; the end-point is quite as sharp as with a smaller antity.

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* American Chemical Journal.

The following titrations show the excess of silver solution required when different quantities of chromate are used, all other conditions being the same:

NaCl sol. taken.	K_2CrO grams.	Vol. of liquid titrated.	$AgNO_3$ sol. required.	Excess.
2 c.c.	·004	50 c.c.	2·45	·45
„	·008	„	2·35	·35
„	·016	„	2·27	·27
„	·030	„	2·20	·20
„	·060	„	2·16	·16
„	·120	„	2·15	·15

The volume of the liquid titrated has also an influence on the result. (The volume may be easily determined, after titration, by pouring the liquid into a graduate.) The required excess of silver solution increases regularly with the volume, and when 0·06 gram. chromate is used, and the volume is not more than 60 c.c., this excess may be represented by the formula $x = \cdot003\,v + \cdot02$, where v is the volume of liquid at the end of the titration in cubic centimeters. Thus with a volume of 3 c.c. the correction is ·03 c.c., while with a volume of 50 c.c. the correction is ·17 c.c.

The following titrations were made in different volumes, all other conditions being the same:

NaCl sol. taken.	K_2CrO_4 used.	Vol. of liquid.	$AgNO_3$ sol. required.	Excess.
2 cc.	·06 gr.	4 cc.	2·03 cc.	·03 cc.
„	„	10	2·05	·05
„	„	20	2·07	·07
„	„	30	2·10	·10
„	„	40	2·14	·14
„	„	50	2·17	·17
„	„	100	2·35	·35

The amount of precipated silver chloride present influences the result. The required excess of silver solution, in addition to the correction for volume, increases regularly with the amount of precipitated silver chloride, and is nearly proportional to the amount of silver solution used. The correction is nearly one per cent. of the volume of the silver solution. The most convenient way to apply this correction is to use a silver solution containing one per cent. more than the theoretical amount of silver. If the silver solution is standardised against sodium chloride, it will be of this strength, and no correction will be required for the amount of precipitate. If the amount of chlorine titrated is very low the results are somewhat irregular, so that if less than 1 c.c. of silver solution is required for 50 c.c. of water, I prefer to run in 1 c.c. of salt solutio and deduct the equivalent of silver solution from the result. The influence of th amount of precipitated silver chloride is shown by the following titrations:

NaCl sol. taken.	K_2CrO_4 used	Vol. of liquid.	$AgNO_3$ sol. required.	Excess.
0·0 cc.	·06 gr.	50 cc.	·10 cc.	·10 cc.
·2	„	„	·30	·10
·6	„	„	·72	·12
1·0	„	„	1·16	·16
2·99	„	„	3.17	·18
5·98	„	„	6·19	·21
9·97	„	„	10·22	·25

The silver solution used for these tritrations was very carefully made to contain exactly the theoretical quantity of silver. A portion of this solution was precipitated with its equivalent of salt solution; to the clear liquid, separated from the precipitate, was added a small amount of salt solution and chromate, and titrated with silver solution. The amount required was the same as when distilled water was used, all other conditions being the same.

Although the original silver solution was made by dissolving the theoretical quantity of pure silver nitrate in water, it is practically more convenient and better to depend upon titrations of salt solution for its strength, always making the correction for volume, As has been shown, a solution made in this way will require no correction for precipitated silver chloride, but the correction for volume must invariably be applied. As this correction is not at all proportional to the amount of silver solution used, accurate results cannot be obtained by using a stronger solution and omitting this correction.

A few titrations of salt solution in varying amounts of water, taken at random from my note-book, are as follows. The silver solution used was standardised against salt solution, and the correction for volume made. In each case ·06 gram. chromate was used.

Milligrams. Cl taken.	Vol. of liquid.	$AgNO_3$ sol. required.	Corrected for volume.	Milligrams. Cl found.
5	16 cc.	10·06	9·99	4·995
5	50	10·18	10·01	5·005
1	40	2·12	1·98	·99
1	70	2·20	1·99	·995
·94	80	2·10	1·84	·92
1·53	46	3·26	3·10	1·55
1·30	30	2·75	2·64	1·32
·87	4	1·76	1·73	·865
2·24	45	4·62	4·47	2·235
9·20	65	18·70	18·48	9·24
3·76	22	7·68	7·59	3·795
·46	60	1·10	·90	·45
3·15	35	6·46	6·34	3·17
1·65	50	3·45	3·27	1·635

It is possible, by direct titration in this way, to get results within ·05 part chlorine per 100,000 in clear waters, when the chlorine is not higher than 10 parts. When the chlorine is low and greater accuracy is required, the water must be concentrated before titration. When this is done, there must invariably be added an excess of sodium carbonate before evaporation, as otherwise there may be a serious loss of hydrochloric acid, even if the residue is not allowed to go to dryness. Commonly 0·02 gram. of sodium carbonate, or less, is sufficient, and this quantity does not in the least interfere with the titration. If too much has been used, calcium nitrate may be added before titration. An excess of nitrate does not influence the result.

I have concentrated waters in three ways, any one of which gives perfectly reliable results. First, by boiling down 500 c.c. or more in a flask with sodium carbonate, to about 50 c.c. By this method there is no possibility of hydrochloric acid being absorbed from the air. It is very convenient, and gives good results when there is not too much organic matter present. In such cases the residue becomes too brown for sharp titration, and must be decolorised by aluminium hydrate and filtered.

Second, by evaporation on a water-bath with sodium carbonate, to a smal

Third, by treating the solid residue of evaporation. All determinations in this laboratory are made with the addition of sodium carbonate, and the i made in a radiator. I have found by a large number of determinations tha no loss of chlorine whatever by igniting in this way, while all organic matter is After the residue is weighed, the chlorides may be extracted with hot water, ferred to a porcelain dish and titrated. When much organic matter i this seems to be the most accurate method, and in all cases it is very convenie must be taken that no hydrochloric acid is absorbed during evaporation, an chlorine is washed from the residue.

The following determinations are given as illustrations of the different and the correction for volume :—

Serial No.	Amount taken.	Vol. of liquid.	NaCl sol. used.	AgNO$_3$ sol. required.	AgNO$_3$ corrected for vol.	Parts per 100,000.	Remarks.
		55	10	10·20	10·02		Standardisation.
2691	50	53	1 c.c.	1·30	·12	·12	Direct titration.
,,	500	45	0	1·53	1·38	·138	Boiled down in a f
,,	250	20	0	·80	·72	·144	Evaporated on a w
,,	250	15	0	·74	·68	·136	Solid residue.
2689	50	53	1	1·35	·17	·17	Direct titration.
,,	500	50	0	1·65	1·48	·148	Boiled down in a f
,,	250	20	0	·80	·72	·144	Evaporated on a b
,,	200	15	0	·65	·59	·147	Solid residue.
2794	50	53	1	1·50	·32	·32	Direct titration.
,,	100	15	0	·75	·68	·34	Solid residue.
2809	50	56	0	5·40	5·21	5·21	Direct titration.
,,	100	30	0	10·55	10·44	5·22	Solid residue.
2821	50	58	0	7·53	7·34	7·34	Direct titration.
,,	50	20	0	7·38	7·30	7·30	Solid residue.

No. 2691 is a sample of the city water of Lawrence; No. 2689 is a sam water filtered through sand; Nos. 2794 and 2809 are other clear waters, and is a sample of sewage.

I have made duplicate determinations of chlorine by two or more of thes in several hundred samples of water and sewage, with exceedingly regular r there appears to be no source of constant error.

Lawrence Experiment Station, Mass. State Board of Health.

DYNAMICAL THEORY OF ALBUMINOID AMMONIA.

By Robert B. Warder.

(*Continued from page* 216.)

Curves of similar properties express the progress of other reactions w place during concentration in large excess of reagents. Equations of the forms are obtained by varying the exponent of x in equation (3) and *appropriate constants.* For reactions involving the concurrence of two molec

$$-\log\frac{z_\infty-(y+z)}{z_\infty}=-A\,.\log\frac{z}{x_0}. \tag{6}$$

For three molecules,

$$-\log\frac{z_\infty-(y+z)}{z_\infty}=A\,\frac{x_0-x}{x}. \tag{7}$$

For four molecules,

$$-\log\frac{z_\infty-(y+z)}{z_\infty}=A\,\frac{x_0^2-x^2}{x_2}. \tag{8}$$

In all such cases the formation of ammonia may take place most rapidly in the early, middle, or latter part of the distillation, according to the value of A.

If the ammonia, instead of appearing directly, is the final product of a succession of different reactions, a complete theory must take account of the gradual formation and disappearance of the intermediate products. Some evidence on this point will appear in section VI.

IV. *Distillation of Albuminoid Ammonia.*

Equation (1) applies to the distillation of ammonia only when the total quantity $(y+z)$ remains constant; otherwise we must write $-dz$ in place of dy. Thus,

$$\frac{-dz}{dx}=k\,.\frac{y}{x}, \tag{9}$$

or

$$y=-\frac{x}{k}\cdot\frac{dz}{dx}. \tag{10}$$

Substituting in equation (7),

$$-\log\left(1+\frac{xdz}{kz_\infty dx}-\frac{z}{z_\infty}\right)=A\,\frac{x_0-x}{x}. \tag{11}$$

This equation (as soon as a fixed value is given to A) expresses a perfectly definite relation between the ammonia distilled and the volume remaining in the retort. It is not easy, however, to obtain an integral in form suitable for computation, and an indirect method was used. The ordinates of curve B represent the sum of y and z when $A=1$; these ordinates were divided into two parts, in accordance with the condition implied in equation (9). Curve D thus represents the gradual increase of z, and curve E shows the increase and subsequent decrease of y. A few corresponding points of these three curves are also indicated in the following table, increments of z being placed under the symbol Δz. These increment are shown on the figure as d, d, d. For convenience, z_∞ is taken as unit of weight for ammonia, and x_0 is made equal to 8; as when a volume of 400 c.c. is distilled in fractions of 50 c.c. each. The ratio of each increment to the next is stated under r.

x.	$y+z$.	y.	z.	Δz.	r.
8	·000	·000	·000	..	..
7	·280	·129	·151	·151	·58
6	·535	·122	·413	·262	1·05
5	·749	·087	·662	·249	1·31
4	·900	·047	·853	·191	1·74
3	·978	·015	·963	·110	3·23
2	·999	·002	·997	·034	..
0	1·000	·000	1·000	..	..

When $A=1$ (as in this table), y reaches a maximum when $x=6·7$, during the collection of the second fraction of distillate; and this fraction contains the largest quantity of ammonia. The third fraction contains nearly the same amount, but later increments show a rapid decrease. The large increase of ammonia indicated for the second measure is due in very small part to the increased concentration of the perm ganate, but chiefly to the fact that sufficient time has elapsed for the accumulation ammonia in the retort. An increase in the second measure has been rarely observed practice, except in a few experiments on pure chemicals, such as urea, theine, glychoch acid, and notably in cyanuric acid. In the 59 samples of natural waters already refer to, the first measure always contained as much as 1⅓ times that found in the second; 11 instances the ratio is 2, and in 33 instances it is more than 2. On the other ha instead of an increase in this ratio for succeeding portions (as indicated by theory fc single reaction), it usually decreases, the ratio of the last two determinations being fı 1 to 2. This want of agreement between the theory for a single reaction and obseı tions on a natural water are exactly what we should expect; for the water may cont a great number of different substances, each undergoing change according to equat (6), (7), or (8), so that the actual rate of distillation depends upon the sum of ammonia derived from all these sources. As curve E shows a rapid rise from zero maximum, followed by a gradual decrease to zero, so we may conceive a multitud similar curves, with their several maxima at various points, but with a large share of tl near the origin; implying $A > 1$, for the most part. The resultant of all such cu might have a single maximum, like the type given, or it might have a successio maxima and minima. On the theory of averages, the former seems more proba This resultant curve might have a great variety of forms in details, answering to various behaviour of different waters.

V. *Exterpolation Formula for Residue in Retort.*

Many chemists, feeling that the task is not completed by nesslerising three measu have pushed the distillation much farther. Mallet suggests a certain limit as criteı when the distillation has been carried far enough. If the ammonia found in the cessive portions is reported, we can rudely plat the curves for ammonia distilled, monia in retort, or total ammonia formed. Each of these curves may be the result of many similar curves having different parameters. Can we find an empirical equat for the resultant that will conform to the analytical results, and thus enable us to ap a probable correction for the ammonia that would be obtained if the distillation w completed?

We may assume a curve of the form

$$y = Ax + Bx^2 \qquad ($$

to indicate the varying quantity of ammonia in the retort under the condition that is all distilled away when the fluid is gone. Substituting in equation (9),

$$\frac{-dz}{dx} = kA + kBx.$$

Integrating between the limits z_1 and z_2,

$$z_2 - z_1 = kA(x_1 - x_2) + \tfrac{1}{2}kB(x_1^2 - x_2^2);$$

or, for brevity,

$$\Delta_1 z = kA\Delta_1 x + \tfrac{1}{2}kB\Delta_1 x^2. \qquad ($$

In like manner, for a second increment of z and for the undetermined portion,

$$\Delta_2 z = kA\Delta_2 x + \tfrac{1}{2}kB\Delta_2 x^2,$$
$$\Delta_\infty z = kA\Delta_\infty x + \tfrac{1}{2}kB\Delta_\infty x_2. \qquad ($$

Those who follow Wanklyn's directions for 500 c.c. of water begin the distilla of albuminoid ammonia with 350 c.c. of fluid, and distil three measures of 50 c.c. e rejecting the last 200 c.c. Taking 50 c.c. as unit of volume, $x_1 = 6$, $x_2 = 5$, and $x_3 =$ while $\Delta_1 z$ and $\Delta_2 z$ represent the ammonia found in the second and third measu Assume $z = r\Delta_2 z$, then

$$r\Delta_2 z = kA(6-5) + \tfrac{1}{2}kB(36-25) = kA + 5{\cdot}5kB,$$
$$\Delta_2 z = kA(5-4) + \tfrac{1}{2}kB(25-16) = kA + 4{\cdot}5kB.$$

By elimination,

$$kB = (r-1)\Delta_2 z \text{ and } kA = (5{\cdot}5 - 4{\cdot}5r)\Delta_2 z.$$

But

$$\Delta_\infty z = 4kA + 8kB,$$
$$\therefore\ \Delta_\infty z = (14-10r)\Delta_2 z. \qquad (15)$$

If

$$r=1,\ \Delta_\infty z = 4\Delta_2 z\,;$$

that is, if the second and third measures yield like quantities of ammonia, the best correction we can make is to assume that the distillation of four times 50 c.c. still remaining in the retort would yield four times that quantity.

If $r > 1\frac{2}{9}$, the value of A becomes negative, and therefore y becomes negative for small values of x. This involves a physical impossibility, but we may meet the difficulty by treating the equation

$$y = Ax^2 + Bx^3 \qquad (16)$$

in the same manner as equation (13), whence

$$\Delta_\infty z = \frac{188-100r}{37}\Delta_2 z, \qquad (17)$$

a correction which again fails if $r > \frac{91}{61}$.*

We may test these formulas by means of Smart's experiments with pure chemicals; but, as we began the action with 400 c.c. of fluid instead of 350, $\Delta_1 z$ and $\Delta_2 z$ represent his third and fourth fractions, with the advantage of greater distance from the beginning of the operation, where equation (12) is wholly inapplicable. The available data (for values of r between 1 and 1·5) are given in the following table, which states under:—

A, per cent. ammonia found in the first four fractions;
B, per cent. ammonia found in "exhaustive experiments";
C, per cent. ammonia obtainable after the first four fractions; and
D, per cent. ammonia indicated by the exterpolation formulas.

	A.	B.	C.	D.
Silver and potassium cyanide, ...	2·25	[17·1]	14·85	2·4
Prussian blue, ...	·3	25·8	52·5	·2
Urea, from urine	8·7	43·10	34·4	8·8
	8·65	..	34·45	6·6
Urea, from ammonium cyanate, ...	7·7	43·4	35·7	8·0
	7·5	..	35·9	8·0
Alloxan, ...	6·55	21·3	14·75	2·5
	6·6	..	14·7	2·4
Thiocarbanilide	4·15	11·85	7·7	1·6
Thiocarbamide, ...	15·25	22·50	7·25	1·7
	15·0	..	7·5	1·6
Strychnine sulphate, ...	4·0	5·4	1·4	·4
	3·75	..	1·65	1·0
Codeine, ...	5·0	5·32	·32	·5

---

* To obtain a more general formula for the correction proposed, we may assume

$$y = Ax^n + Bx^{n+1}, \qquad (18)$$

whence, by the same treatment illustrated above,

$$\Delta_\infty z = \frac{(\Delta_1 x^{n+1} - r\Delta_2 x^{n+1})\Delta_\infty x^n - (\Delta_1 x^n - r\Delta_2 x^n)\Delta_\infty x^{n+1}}{\Delta_2 x^n \Delta_1 x^{n+1} - \Delta_1 x^n \Delta_2 x^{n+1}}\Delta_2 z, \qquad (19)$$

where A is positive or zero when

$$r \leqq \frac{\Delta_1 x^{n+1}}{\Delta_2 x^{n+1}}. \qquad (20)$$

We may either make n the smallest integer consistent with the condition (20), or we may determine n by making the fraction equal to r. The function $\Delta_\infty z = \phi(r)$ will be a smooth curve in either case, consistent with the theory as developed above and with the analytical results.

A glance at the last two columns shows that the ammonia actually obtainable aft one-half of the fluid had been distilled was many times the proposed correction. A explanation of this discrepancy will next be considered.

VI. *Evidence of Intermediate Compounds of Nitrogen.*

Making some allowance for the difference between equation (7) (which was selecte for study) and equations (6) and (8), it is safe to conclude from the table in se tion IV, that if the action of alkaline permanganate upon a pure compound nitrogen could be represented by any single chemical equation (no matter how complex we should have reason to expect this change to take place with such speed that full 70 per cent. of the theoretical yield of ammonia would be obtained by distilling one-ha the fluid, or else with such slowness that the first measure would contain far le ammonia than the second. Published experiments with at least eight bodies* contr dict both these conditions, and we are forced to believe that in the earlier stages som molecules of the original body yield ammonia with promptness, while others are co verted into more stable forms and resist the action with great stubbornness. Th nature of these intermediate products is not yet clear, except only that a (as defined i section III.) is small, in their reaction with alkaline permanganate. Perhaps they may b chiefly condensation products. Alloxan, which yielded 4 per cent. ammonia in the fir fraction (eighth) of distillate, and only 2·57 per cent. in the next three, but 21·3 pe cent. by prolonged action, is a marked example of such behaviour; this, no doubt, partly converted into parabanic acid. So, likewise, in a water analysis, many substanc may yield a small part of their nitrogen as ammonia, while the greater part is converte into more stable forms which elude detection. It has been supposed that the organi matter of potable water is more like asparagine, yielding its nitrogen readily under th action of permanganate. Of this, however, I have found no proof. In about 40 per cen of the natural potable waters referred to, the ratio of the third and fourth fractions i 1·5 or less, giving grave suspicion of incompleted action when the distillation wa stopped. Even if the yield of ammonia in some fraction has fallen below the limit c delicacy in nesslerising, what evidence have we that prolonged treatment would no develop a much larger amount of ammonia than that already obtained?

VII. *Applications to the Analytical Process.*

A debt of gratitude is due to Professor J. Alfred Wanklyn, as the author of valuable method of analysis which has done much in the interest of public health "The ammonia process," which originated in 1867, is still described in the 7th editio of "Water Analysis" (1889) essentially as in the author's first paper upon the subjec In a process of such delicacy, where the results may be influenced by a multitude o variable conditions, it is desirable that these should be accurately defined and conscientiously followed. According to the author's latest directions, 500 c.c. of water ar reduced to 300 c.c. (to remove free ammonia) and 50 c.c. of the reagent added; thre measures of distillate are then collected, when "the distillation may be stopped . . . It is necessary to nesslerise each separate 50 c.c. of distillate, and to add the amount together, in order to arrive at the total albuminoid ammonia." Besides this process, second is described upon a scale of one-fifth, with this important modification, that the operation is "continued so long as 10 c.c. of distillate shows a sign of ammonia"; if, then, with a delicate Nessler reagent, ammonia is found in the sixth measure of distillate, the fluid must be evaporated to dryness!

---

* These are the first six named in the last table, besides parabanic acid and theine.

But why should 350 c.c. be distilled until only $\frac{3}{7}$ have gone over, while 70 c.c. are distilled up to a very different limit? Probably because the *time* required to evaporate 30 c.c. from the smaller retort proved insufficient for both the chemical action required and for the separation of the ammonia. Professor Wanklyn's method, as conducted in his own laboratory and by his own hands, has doubtless done valuable service, yielding good comparable results; but more definite conditions must be adopted if we would avoid discrepancies among different places and persons. Its value depends upon giving constant and comparable results under fixed conditions. For the absolute estimation of organic nitrogen, it is worthless.

It will be instructive to divide the free or albuminoid ammonia found in each measure of distillate by that found in the next, and to record the ratio so found; especially if care is taken to boil at a constant rate. According to Smart,* with pure animal or vegetable albuminoids, $r=2$; but if these have passed into the putrefactive condition, $r=3$; while in the case of urea (after the preliminary stage is passed) $r=1$, both for free and for albuminoid ammonia. The successful detection of recent sewage is reported, when the chemical evidence consisted in the persistent evolution of free ammonia, followed by persistent evolution of twice as much albuminoid ammonia.

The term "albuminoid ammonia," like "reverted phosphoric acid," has a technical significance determined by the method adopted in the analysis. The actual market value of reverted phosphoric acid cannot be so high under the rule for extraction at 65° as under the rule for extraction at 40°; so also 0·1 mgm. of ammonia obtained by prolonged and intense action is of less real importance than the same quantity obtained by less vigorous measures. Many modifications of the original process have been proposed; among these Breneman's† are worthy of special consideration. If the fluid is brought nearly to dryness, as he suggests, it may be possible to apply an exterpolation formula, such as equation (19), to the measured residue. The detailed record of ammonia found in seven successive fractions (with greatly increased temperature and concentration at the last) may help to distinguish between different kinds of nitrogenous matter, yielding ammonia more readily and less readily; but care should be used in regard to the volume of the residue; if this is 4 c.c. in one case and 7 c.c. in another, there may be considerable difference in the oxidation of that comparatively inert matter which we have been led to suspect.

If the ammonia process should be applied to investigations of the relative stability of organic compounds,‡ the formulas given above may be adapted to numerous possible modifications. The use of return-flow condensor (to prolong the chemical action apart from distillation), and variations in the proportions of potash and permanganate, do not seem to have yet been tried. It is difficult to see why the alkali should always be used in so much greater excess than the oxidiser (in spite of inconvenient bumping), but it is especially strange that the distinguished author, ignoring Mallet's report of 1882, and republishing his directions without any revision, should leave the important question of *time* entirely out of account. "Suffering persecution for the sake of the truth" should make the scientist keenly alive to any truthful criticism, and ready to study the contributions of his colleagues. The "ammonia process" has now passed from one laboratory into a hundred. If "discovered about a generation before chemists were prepared for it," the time is now fully ripe to collect the testimony of those who have used it; to agree upon the general conditions of quantity, rate of boiling, and limit of distillation, and even upon such details as the matter of paper or rubber connections. When a satisfactory compromise is effected we may hope to have a uniform method of procedure and a real definition of albuminoid ammonia.

---

* Public Health (Minn.), Vol. II, No. 3, May, 1886.

† Jour. Amer. Chem. Soc. 8, No. 9.

‡ It may be of interest to compare such results with those of Dreyfus, who studied the rate of oxidation with acid solution of permanganate Compt. rend. 105, 523.

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

DEXTRIN IN GENUINE HONEY. C. AMTHOR AND J. STERN. *Zeitschr. f. angew Chemie*, No. 20, 1889.—44·9655 grms. of a sample of undoubtedly genuine honey were dissolved in 300 c.c. of water and allowed to ferment with yeast. After filtering and making up to 200 c.c., the liquid was examined in a Laurent's polariscope, and gave +24·9° in a 20 c.m. tube. The dextrin was then converted into dextrose, by heating 100 c.c. of the liquid with 10 c.c. of hydrochloric acid for three hours in a water-bath. After cooling and making up again to 100 c.c., the polarisation gave 9·21°, equal to 2·249 per cent. of dextrose. Allihn's gravimetric sugar process gave 2·266 per cent. The formation of dextrose points to the honey having contained dextrin. The authors further confirmed this view by preparing phenylglycosone. A large quantity of honey was fermented in a weak solution. The filtrate yielded on evaporation a brown thickish syrup, which was really an impure dextrin. After dissolving in a little water, phosphotungstic acid, containing some sulphuric acid, was added, and the liquid filtered. The excess of the reagent was removed by means of baryta water, and this in turn removed by cautious addition of sulphuric acid. After concentrating a little, liquor plumbi was added, the liquid filtered, and the excess of lead removed by hydrogen sulphide.

The filtrate was evaporated in the water-bath until it became a thin syrup, and 200 c.c. of 90 per cent. alcohol were added, which was sufficient to keep the dextrin in solution. After filtering and evaporating, the dextrin was obtained as a brown syrup, which was purified with animal charcoal, until it was finally got as a yellowish brittle amorphous mass. It is exceedingly soluble in water, very hygroscopic, soluble in spirits of wine, but insoluble in absolute alcohol or ether. The watery solution very slightly reduces Fehling's liquid.

5 grms. of this dextrin were dissolved in 200 c.c. of water, mixed with 20 c.c. of 20 per cent. sulphuric acid, and heated for three hours in water-bath. The liquid was neutralised with barium carbonate, filtered, and evaporated to a syrup, which was

epeatedly exhausted with alcohol. After distilling off a brownish syrup was obtained, hich was purified by animal charcoal.

It crystallises in granules, reduces Fehling's strongly, and the authors succeeded in reparing phenylglycoson, by warming 1 grm. with 2 grms. of phenylhydrozin, 3 grms. of odium acetate, and 20 c.c. of water. The crude product was recrystallised from alcohol nd obtained in small yellow needles, fusing at 204° C. This removes all doubt about he original substance being dextrin, the presence of which in honey may perhaps be xplained by the bees feeding, when they get the chance, on malt in breweries.

L. DE. K.

TREATMENT OF URANIUM RESIDUES. D. LAUBE. *Zeitschr. f. angew Chemie,* o. 20, 1889.—The liquids resulting from the precipitation of phospates with uranium lts are kept in a large vessel, and the supernatant fluid is occasionally drawn off. Vhen a sufficiency of precipitate has collected, the whole is heated to boiling in an iron essel, and common soda is added until the precipitate seems dissolved. After cooling, nd without filtering, ammonia is added until the mixture smells perceptibly of it, and the hosphoric acid is then thrown down with magnesia mixture, prepared by dissolving equal uantities of magnesium and ammonium sulphates. After, say, twelve hours, the liquid s syphoned off, and the residue washed with ammoniacal water. The united filtrates re now neutralised, either with hydrochloric or sulphuric acids, and freed from carbonic cid by boiling. The uranium is now precipitated by ammonia, as uranic-ammonic xide. This precipitate is washed by decantation, but it is advisable to put a small uantity of ammonium sulphate to the wash water.

By dissolving the precipitate in excess of nitric acid and evaporating, commercially ure uranic nitrate is obtained in crystals, which are allowed to drain on a funnel and vashed with a very little cold water.

L. DE K.

ESTIMATION OF TANNIC ACID IN OAK BARK, WITH PERMANGANATE. F. GANTTER. *eitschr. f. angew Chemie,* No. 20, 1889.—The author found that tannin is completely nd *quantitatively* oxidised by permanganate when its solution is mixed with dilute ulphuric acid and boiled. To estimate the tannin in a sample of oak bark, the following olutions are wanted: 1. The usual bark decoction, 10 grms. per litre. 2. A solution f permanganate, containing 3·988 grms. of $K_2 Mn_2 O_8$ per litre. 3. A solution con-ining 7·951 grms. of oxalic acid per litre. 10 c.c. of the bark solution are mixed ith 10 c.c. of dilute sulphuric acid, and heated to nearly boiling. Permanganate is ow added, about 1 c.c. at the time, until the red colour but slowly disappears. The uid is now once more heated to boiling, and permanganate is *gradually* added in cess, which causes a heavy brown precipitate. Oxalic acid is now run in until the uid has become quite clear, and permanganate is then again added until red colour is stored. The total number of c.c.'s of permanganate used, less those of oxalic acid, is e amount reduced by the tannin, and at once represents the percentage of tannin in e sample.

It is of course advisable to repeat experiment after removal of tannin by hide wder as usual. The author is now engaged in trying to find out the exact relation tween permanganate and the other kinds of tannic acid.

L. DE K.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

MILK ANALYSIS.

DEAR SIR,—In an article on "Milk Analysis" in the ANALYST of November, 1889, Dr. B. F. Davenport states that the fat can be *entirely* extracted from the dried residue of 5 grms. of milk, if only this be in a flat-bottomed platinum dish of 2½ inches diameter. He uses four separate quantities of boiling benzoline. Unluckily, he gives no comparative analyses to show that the fat is *entirely* extracted.

For years I used exactly the method described, sometimes with larger flat-bottomed dishes, and always with more numerous changes of benzoline, boiled on the residue. The fat, however, was never entirely separated, nor even to within "one tenth of a per cent." In a paper in the February number of the ANALYST, 1889, he will see this method contrasted with more modern methods. In my experience, there are only four methods that extract all the fat from a milk. 1st. Soxhlet's Areometric method. 2nd. Soxhlet's tube with pumice, plaster of Paris, kieslguhr, or paper-coil (Adams). 3rd. The Lactocrite. 4th. The Schmid method; this last was described in the *Chemical News*, of November 1st, 1889.

Yours faithfully,
ALF. W. STOKES, F.C.S. F.I.C.

Analytical Laboratory,
Vestry Hall, Harrow Road,
November 4th, 1889.

To the Editor of the ANALYST.

SIR,—I have read with extreme interest, Dr. Bennett F. Davenport's article upon Milk Analysis, as printed in your last issue. I thoroughly agree with him in all that he has written concerning the estimation of the fat in milk, the process employed, being termed by him a modified Wanklyn. I am in the habit of using platinum basins of 2½ inch diameter, but having quite flat bottoms, and have no difficulty whatever in obtaining a very thin and evenly distributed skin of residue, the complete extraction of which by means of petroleum ether, is easily accomplished. Petroleum ether as mentioned by Dr. Davenport, having a great advantage over that of ether in not dissolving either lactic acid or lactose. The process known as the "Adams blotting paper," and recommended by the Milk Committee of the Society of Public Analysts, is a process which I consider to be faulty, and that in three respects, and my reasons for saying so, are as follows:—

1st. You cannot estimate directly both the fat and the solids not fat. 2nd. That the amount of fat obtained is in excess of that originally present in the milk, owing to an increase in weight, due to rapid oxidation of the fat when in contact with cellulose, when spread over such a large surface, and also to a further oxidation which takes place when completing the drying of the extracted fat in the water oven. 3rd. It cannot be used when the milk is not fresh, objections which are, in my opinion, fatal to any milk process.

I used blotting paper in milk analysis in 1882 and relinquished its use owing to the last objection, and finally by a long series of experiments, satisfied myself as to the correctness of my second objection, and when coupling that with the first and the third objection, I have now no hesitation in rejecting it as faulty.

My opponents may say that the fat calculated from the sp. gr. and total solids, agree with that found by Mr. Adams' process, but to that I reply, we have already had examples of altering formula to suit the particular process employed.

I am, yours truly,
WILLIAM JOHNSTONE.

City Central Laboratory,
13, Fish Street Hill, E.C.
November 4th, 1889.

APPOINTMENT.

Mr. J. Falconer King, Analyst to the City of Edinburgh, has been appointed Public Analyst for the County of Haddington.

ERRATUM.—On page 217, vol. xiv., line 2, after 1889, read "and *Jour. Soc. Chem. Industry*, September 1889."

The Analyst,

INCLUDING THE PROCEEDINGS OF

THE "SOCIETY OF PUBLIC ANALYSTS."

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT OF THE
ANALYSIS OF FOOD AND DRUGS, AND OF GENERAL
ANALYTICAL AND MICROSCOPICAL RESEARCH.

EDITED BY

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VOL. XV.

1890.

LONDON:
PUBLISHED FOR THE PROPRIETOR, BY MESSRS. BAILLIERE, TINDALL & COX,
KING WILLIAM STREET, STRAND, W.C

- 19015 -

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HAZELL, WATSON, & VINEY, Ld.,
London and Aylesbury.

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THE ANALYST.

JANUARY, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of the Society was held at Burlington House, on Wednesday, the 11th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

A ballot was taken for new members, and the following were declared to be duly elected:—As members, C. H. Cribb, B.Sc.Lond., F.I.C., F.C.S., public analyst for the Strand; W. Hepworth Collins, F.C.S., analytical chemist, Bolton. As associate, S. H. Collins, assistant to Mr. Young.

The following gentlemen were proposed for election:—W. Newton, Ph.D., F.I.C., F.C.S., analytical chemist, London; G. H. Masson, of Trinidad.

Mr. Turner and Mr. Cassal were appointed as auditors, to audit the accounts for the past year.

The adjourned discussion on Mr. Allen's paper on "Possible Future Extensions of the Work of Public Analysts," was then resumed and concluded.

The Annual Meeting of the Society will be held at Burlington House, on Wednesday, the 8th inst., and followed by the annual dinner, as usual.

THE POSSIBLE FUTURE EXTENSION OF THE DUTIES OF PUBLIC ANALYSTS.

By Alfred H. Allen.

(*Read at the Meeting, November*, 1889.)

In my Presidential Address, delivered at the annual meeting in January last, I called attention to the possibility of enlarging the sphere of action of public analysts. At present the duties of public analysts are well known to be restricted to the examination of food and drugs, and owing to the clause relating to the examination of articles purchased by private persons having proved nearly inoperative, a public analyst's duties are at present almost wholly limited to the examination of samples purchased for the purpose of analysis by inspectors acting officially. Even the analysis of water does not come within the official duty of public analysts, owing, as we are well aware, to the exclusion of drinking water from the operation of the Sale of Food and Drugs Act.

Owing to the publication of improved methods of analysis, to which the Society of Public Analysts has so much contributed, and to other causes with which we are all familiar, food analysis has acquired a position which none of us could have anticipated sixteen years ago. At the present time, the public analysts of the country are a body of highly competent men, whose special training, not only as analysts but as chemico-legal experts, specially qualifies them for executing the responsible duties of State chemists, and there are many new directions in which the public analyst might be employed to the advantage of the public.

It seems to me a grave public evil that the provisions of the Sale of Food and Drugs Act are not extended to include articles which do not come under the denomination either of a food or a drug. Thus, for instance, there have been successful prosecutions in some parts of the country for the sale of adulterated *olive oil*; but it seems to me doubtful whether olive oil comes under the Act, unless it is intended for use as a food or a remedy. That it is an article named in the British Pharmacopœia is not sufficient, for, unfortunately, when the Sale of Food and Drugs Act Amendment Bill was in committee in 1879, Dr. Bell advised that the British Pharmacopœia should not be formally adopted as the legal standard of all articles described therein. He did this on the ground of non-necessity, as the Pharmacopœia was already substantially regarded as the authority; but we all know how far this is from being the case in practice. Partly, this may be due to the very inefficient manner in which parts of the Pharmacopœia are edited; but if in a new Act the Pharmacopœia were made the legal Standard for drugs, such pressure would be brought to bear on the editors as would compel them to remove the blemishes which at present discredit their work.

It is very questionable, again, whether *yeast* can fairly be regarded as coming within the four corners of the existing Act, though any new Act should certainly be drafted so as to include it.

Carbolic disinfecting powders, again, are articles which are by no means always of the quality alleged. Corporations and Boards of Health suffer largely from deficient strength in the carbolic powders supplied to them; a powder sold as containing 10 per cent. of tar acids in reality often contains only 6 or 7 per cent., and sometimes as little as 4 per cent., and the members of the general public are also very liable to get very poor value for their money and disinfectants of doubtful strength and efficacy.

Petroleum, again, is an article which might, with advantage, be occasionally submitted to public analysts. The Petroleum Act requires no alteration for this to be done, as it provides for the testing of the oil by the inspector "or other competent person selected by him," which person should be the public analyst for the district.

There ought also to be some definite control over the sale of articles containing *arsenic*, such as carpets and paper-hangings. If an Act were passed on the lines of the Margarine Act, enabling an inspector to visit shops were carpets and paper-hangings were sold, and to take for examination by the public analyst samples of any goods which were not *marked* as containing arsenic, the public would know what they were buying, and arsenical colours would soon be a thing of the past.

The same principle might be applied to *mixed fabrics*. All mixed fabrics should be labelled as such, the responsibility of labelling them to rest with the vendor.

I think that the inspectors under the Sale of Food and Drugs Act might be advantageously employed to see that the provisions of the Pharmacy Act are carried out, instead of leaving the matter, as at present, to the Pharmaceutical Society. The inspector should be instructed to ask for a *poison*, and see that the sale was properly recorded, and the name and address of the vendor entered in the Poisons Book.

It would also be a great advantage if it were enacted that all poisons sold as *vermin-killers* should be coloured with a definite proportion of some colouring matter, which could be easily detected and estimated. A few months since a man in Derbyshire poisoned his child with vermin-killer containing strychnine for the sake of the insurance money, and would not have been convicted and hanged but for the presence of a blue colouring matter in the vermin-killer. In connection with this case I received from the police a number of packets of vermin-killers containing strychnine, and I supplemented these by ten other samples, representing the chief wholesale manufacturers in the kingdom. The results of the analysis of these samples showed that ultramarine was the colouring-matter most commonly used, though Prussian blue occurred in several cases. Carmine was also present in several, and one specimen was wholly colourless, consisting merely of a mixture of strychnine and starch. In two other cases the colouring-matter was only sufficient to tinge them a very pale flesh or pink colour. One sample only was coloured with soot. It seems to be very important that vermin-killers should be so coloured as to preclude the chance of their being taken accidently by human beings, and so as to facilitate their detection in cases where they have been swallowed, whether they may have been used for the purpose of suicide or murder.

At present, *arsenic*, if sold in quantities of less than 10 lbs., has, by law, to be coloured with soot or indigo, though it is a fact worthy of note that the solid arsenic found in the house of the late Mr. Maybrick was all coloured with wood-charcoal. Carbon, in any form, is not a suitable colouring-matter, as, in small quantities, and in presence of miscellaneous organic matter, it is difficult to identify, and indigo is too susceptible to oxidising and reducing agents to render it a desirable pigment. Prussian blue and ultramarine each have their disadvantages, and a mixture of indigo, Prussian blue and ultramarine would be better than either of these colouring-matters separately.

On the whole, perhaps, the most suitable pigment for colouring vermin-killers would be the oxide of chromium. In it we have a bright green pigment of high colouring power, quite insoluble in water and in dilute acid and alkaline liquids. It is wholly permanent under all imaginable conditions, and is not affected by ignition. Chromium is not a natural constituent of the body, is not used internally as a medicine, and is not liable to be present accidently, even in traces, in any beverage or article of food. It can be detected and determined with ease and certainty, even when present in very minute quantity. Owing to its insolubility, oxide of chromium would remain wholly unabsorbed if taken internally. Hence, if it were added to preparations of strychnine in a definite and invariable proportion, say 20 per cent., an estimate of the *minimum* amount of poison taken by the deceased person] could be arrived at by determining the quantity of chromium contained in the ailmentary canal, even though the poison itself had been wholly absorbed or decomposed; and this could be effected with equal ease and certainty after prolonged inhumation, or even after cremation of the body.

But it would be useless to enact that all preparations of arsenic, strychnine, and other deadly poisons sold as vermin-killers should be coloured with a definite proportion of mineral pigment, if the sale of fly-papers, containing a dangerous or fatal dose of arsenic or other poisons, is to be permitted with impunity. All such fly-papers should be impregnated with some soluble colouring matter, which would colour the water in which they were soaked. But organic colouring matters are less suitable for the purpose in question than certain inorganic colouring matters. Of these the soluble salts of chromium present several advantages. Thus, if in the form of the double tartrate of chromium and potassium (or a mixture of chrome-alum and Rochelle salts) the chromium is not precipitated from its solution by alkalies, acids, sulphuretted hydrogen, sulphide of ammonium, or phosphates, or carbonates of the alkali metals. On the other hand, the chromium will not interfere with the detection and determination of arsenic, strychnine, and other poisons, and can be itself determined with ease and accuracy after evaporating the liquid to dryness and igniting the residue. Hence I hold that it is highly desirable that a law should be passed compelling the colouring of vermin-killers, fly-papers, etc., with an indestructible colouring matter, the quantity of which should bear some definite proportion to the active poison present; and I think pnblic analysts might be advantageously employed to see that such a law, when passed, was enforced.

The directions indicated are only some of those in which it seems to me public analysts ought to look in the future for the extension of their duties. In fact, a public analyst of the future ought to become a State chemist, who should be officially consulted on all subjects within his province. There is much to be done before we can look forward to public analysts occupying such positions; but this is a greater reason for endeavouring to promote our ends. Personally, I have alluded to the desirability of extending the present acts relating to sanitary chemistry in several of my quarterly reports, and I believe some few other analysts have done the same, but I am afraid many public analysts have failed to recognise, or at least to use, the opportunity afforded by these quarterly reports of ventilating subjects of the kind.

We may accept it as certain that during the next Session of Parliament the Government will initiate some new legislation dealing with the sale of poisons; and the occasion may serve as an opportunity for urging on them the importance of making some modification in the Sale of Food and Drugs Act, and other laws relating to sanitary chemistry. It is highly important, therefore, that we should not wait for the Bill dealing with the subject to be drafted by some one who will ignore our existence, as has happened on more than one occasion, but that we should ourselves draft a Bill embodying our views, and which may form the foundation of the legislation we hope for.

That the Society of Public Analysts can do much in the suggested direction is certain, if the members will all pull together and act as an impelling force instead of as a drag on their representatives. The Society has done much in the past, in spite of lukewarm support from many of its members, and more or less open opposition from the outside. The importance of possessing an organisation such as ours has been shown recently in several instances. In short, the Society of Public Analysts, though primarily a scientific society, has other duties to perform, including the guidance of Parliamentary work in connection with the subjects already referred to, and the amendment of the Sale of Food and Drugs Act generally; the discussion of legal points affecting us as public analysts; the assistance of our members in cases in which our common interests are concerned; and in a host of other ways.

DISCUSSION.

Dr. **SEATON** said he thought Mr. Allen's paper was a very valuable and suggestive one as it affected health. The Food and Drugs Act was passed primarily in the interests of health; all the matters touched upon related to health, like colouring-matters, carpets, and so on; and these would seem to be matters that public analysts would be doing good service in reporting upon. Then Mr. Allen had referred to the very important influence they might have in helping to carry out the provisions of the Pharmacy Act. He lately had had to confer with a well-known firm of chemists about a new disinfectant being introduced; it was one that contained a large amount of perchloride of mercury, which, no doubt, could be bought easily, in the same way that vermin-killers could be bought at unauthorised places. There should be some check upon that. Chemists and druggists were bound down by hard and fast rules with regard to the sale of poisons, and other establishments should be watched which are not under the same regulations. The colouring-matter in this case was very distinct, and the remarks which Mr. Allen made with reference to the importance of adopting some colouring-matter which would bear a definite proportion to the poison were, he thought, such as would recommend themselves to others, and of a very practical nature.

Mr. **CASSAL** said that he thought Mr. Allen's paper was exceedingly important from the point of view of the development of public analysts. Such a development was precisely what was, at the present time, most urgently required; but it would be difficult to obtain without the better instruction and enlightenment of the people who were entrusted with the appointing of public analysts and with the enforcement of the laws against adulteration. Mr. Allen's suggestions did not go quite far enough, but, at any rate, before they could be properly adopted the general state of things at present existing would have to undergo considerable alteration. It would be necessary to teach the public that the sum of 10s. 6d. was not the right measure of the value of an analysis; they would have to endeavour to get that most objectionable clause, which, perhaps, more than anything else, had tended to injure public analysts and to lower their *status*, struck out of the Act of Parliament. There could be no question that a great extension of their duties as public analysts would ultimately come about, but the attainment of this result would not be greatly hastened by discussions in their own body which were not followed by some sort of united action. They wanted additional legislation, or, at least, the extension and amendment of the present laws in the necessary direction. As a matter of fact, every public analyst knew that the majority of local authorities looked upon the "analyst" as a sort of necessary nuisance, who was to be paid the smallest possible sum for doing the smallest possible number of samples. This was occasionally varied by the payment of the smallest possible sum for the analysis of the largest possible number of samples. It was certainly the duty of the Society to consider as to the best means of improving the position of the public analyst, but the Society was not powerful enough to do what was required. He could not agree that it was even strong enough to afford really adequate support to public analysts in their struggles and difficulties with local authorities. A more powerful body was necessary for this. The extension of their work in the direction of the examination of disinfectants, water, gas, and so forth, was plainly what they were entitled to expect. All such work should be placed in the hands of public analysts, as official chemists. The examination of gas, for example, was at present carried out in the metropolis by a very limited number of "gas-examiners," who were paid an absurdly small remuneration for the work. In the provinces it was practically not carried out at all. It was true that gas was occasionally examined by medical officers of health, and there was the curious fact that these officers also examined water. This led him to another question, that of combined appointments. It would be necessary to put an end to the combined appointments of public analyst and medical officer; it was a thing which led to abuses of all sorts—the farming out of the public analyst's work to a cheap chemist, or of the medical officer's work to a cheap doctor. The holding of combined appointments would have to be strongly discountenanced and, as far as possible, prevented. The subject was one of very wide scope, and he should be glad to hear the opinions of all his colleagues upon it. The future of public analysts largely depended upon their clear appreciation of the position which they held with regard to the public, and the extent to which they might be able to impress the public mind with the enormous importance and the heavy responsibility of their work. A practical suggestion which he would venture to make was this, that every public analyst, whether he was in close contact with his local authority or not, should take every opportunity of instructing that authority upon matters relating to public analysis, with the main view of getting it to work upon the legislature. By united action of this kind all over the country, it was obvious that considerable effects might be produced, which would not only be valuable from a professional point of view, but would be of the greatest service to the public. He suggested that the discussion should be adjourned, so that they could be prepared at a subsequent meeting to enter into a thorough examination of all the points raised.

Mr. Dyer said that since the Parliamentary Committee was appointed there had been no occasion to call a meeting; they were not appointed with general instructions to force their opinions upon the legislature, but to deal with definite matters that might arise. While Mr. Cassal was speaking he could not help thinking that that gentleman might have given a little more point to the fact that every rule had its exception. Their worthy President was a public analyst, and was also one of the ablest and most energetic medical officers of health in the country, and after what had happened that evening (referring to Mr. Adams's paper on a new form of air-bath), he (Mr. Dyer) thought they had had a very recent illustration of the practical skill and competence of an analyst in a medical officer.

Mr. Cassal said that of course he had not had the slightest intention of casting reflections either upon the President or upon Dr. Seaton. He considered that the existence of these combined appointments was injurious to both professions. Among other things, it resulted in this, that the work of one of the two offices was generally not paid for. Certain local authorities got one man to undertake the work of two distinct offices for the pay which should attach to one of them. It therefore appeared to him to be detrimental to the interests of both public analysts and medical officers of health, that they should hold combined appointments, and he thought the two professions ought to unite in discountenancing them.

After other remarks, and it appearing to be the general opinion of the meeting that the matter should be adjourned, some discussion took place as to the precise terms of the motion, and after various suggestions had been made, it was ultimately resolved, on the proposal of Dr. MUTER, seconded by Dr. JOHNSTONE, "That Mr. Allen's paper be printed and circulated with the agenda for the next meeting, and that the discussion be adjourned until that meeting."

RESUMED DISCUSSION ON MR. ALLEN'S PAPER.

The PRESIDENT said that at the last meeting, after a considerable amount of discussion, it was thought better to adjourn it until that evening, and it seemed to him that it might be conducted under three different headings:—

1. Whether it was desirable to promote the wider scope of public analysts duties?
2. If so, could the Society of Public Analysts wisely take any public action in the matter?
3. Whether the present was a good time to take any such action?

MR. HEHNER, before entering into the discussion, read the following extracts from a letter of Dr. A. Ashby:—

"I have received the proof of Mr. Allen's paper. I hope he is not going to agitate for water to be brought within the scope of the Sale of Food and Drugs Act. If it is, we shall not only have an indefinite amount of water analyses forced upon all those public analysts who are paid by fixed salary, but we shall have the general fees for water analysis levelled down to Dr. ———'s fee as advertised in the *British Medical Journal.* I think it is of immense importance that water should be rigidly excluded from the Act; if included we should be laid open to having much work in that direction thrust upon us for nothing, or next to nothing."

Mr. HEHNER then said, that the President having laid down for discussion three questions, he should like to see them answered by the Society as follows:—1. It is *not* desirable to extend the duties of public analysts. 2. The Society should *not* take any action in the matter, and 3. The present time is *not* opportune should any action be deemed desirable.

The Sale of Food and Drugs Act comprised all food and drugs used by man; the Act could not well be wider than it was at present, unless all commercial articles were included in it. These were at present satisfactorily dealt with by commercial men interested in them, and by analytical chemists generally, of which division of the profession, public analysts were, after all, but a very small part. Public analysts certainly could not fairly agitate to obtain by law, the work which belonged rightly to the analytical chemists of the country. If they did, they would lay themselves open to the charge of being both needy and hungry, and wanting that which did not belong to them. Were for instance, cattle foods brought within the Act, he had no doubt that those analysts who now controlled the cattle food supply of the country in a most able manner, and to the benefit of the public, would decidedly object to having their work, which they had acquired by their own industry, taken away from them. The same observation applied to petroleum, and many other articles of commerce now analysed by private, not public analysts. As regards water, it was only by the exertion of the Society of Public Analysts that it had been specially excepted from the Sale of Food and Drugs Act, to the benefit of analysts generally.

An unavoidable consequence of widening the Act would be a general depression of fees, which were already now, in most cases, utterly unadequate. This state of things had been brought about by the Sale of Food and Drugs Act. That Act stipulated, that a public analyst should, for a fee of half-a-guinea, analyse any article of food for any private individual. The Act intended, that a salary be paid by the appointing authorities to the analyst, to enable him to do the work for so low a fee. The public, as all were aware, had taken no action whatever, appointing authorities hardly ever did pay salaries, and yet the fee, the official and now generally accepted fee, had been brought down to half-a-guinea for each sample, while formerly two guineas were generally obtained. As a set off the number of samples analysed had certainly much increased. It might be said, that there was no need to mention any fee in the proposed new Act, but he contended, that the half-guinea fee had now become so general, it had become so burnt into the mind of the public, that there could be no hope of obtaining a higher fee, in future. As commercial analyst as well as a public analyst, he therefore objected to any extension of the Food and Drugs Act.

The Society certainly should not take any action. The men who were directly interested in this matter could not well agitate for more work; they would only make themselves ridiculous if they did so. If there were a public agitation, and if to meet a public want a new Act were passed, then of course public analysts would be ready and glad enough to obtain more work.

No time could be more inopportune than the present. Parliament was almost at the end of its natural life, and occupied with other mattters. He doubted further, whether any Goverment, Conservative or Liberal, would risk to make as many enemies as the passing of such an Act would be sure to make. All manufacturing classes would be against it. It should be remembered, that when the Adulteration Act was passed, it roused so much opposition to the then Government, that it was one of the main factors which led to its overthrow. The next Government at once appointed a Committee, and the present Food and Drugs Act was the result. No Government would risk to burn its fingers over such a question again.

He hoped, therefore, that the Society would meet the three questions with a distinct negative. Yet he thought that the subject was one which should be fully discussed by public analysts.

Mr. Cassal agreed with Mr. Hehner in thinking that the present time was inopportune for taking action, and that it was undesirable for the Society to move in the matter in any official sense, at least at present, but he could not agree with him in the view that an extension of official public analysis was undesirable. He considered that it was desirable in the first place for the benefit of the public, and desirable also for professional reasons and in the interests of both public and private analysts. He could well understand that some members of the profession engaged in commercial analysis should, at first sight, view the extension of official analysis to other articles besides "foods and drugs" with disfavour, but he did not think that they would do so after fuller consideration. The public did not appreciate the value of analytical work. They were ignorant, and could hardly be expected to do so. The inadequate manner in which analysts were paid was largely due to want of proper appreciation of the value of the services rendered, and to a want of proper respect for the professional man who rendered them—in fact, to public ignorance. His belief was that an extension of official work would tend to educate the general public more than anything else. It would gradually show them the great importance of the work, and accordingly would lead to a better appreciation of those who did it. As to the 10s. 6d. fee, he had stated on the last occasion that before any extension of the duties of public analysts was brought about, it would be necessary to destroy the commonly-accepted and highly-convenient notion that this sum was the proper measure of the value of an analysis. If this could not be done, and if it was to be officially laid down that such a fee was a proper one for any analysis whatever, then he agreed with Mr. Hehner that any extension was very undesirable. But it was necessary to make local authorities and the public, clearly understand the facts about the 10s. 6d. fee. It was a nominal fee, intended to bring the services of an officer in the receipt of a proper salary, and in consideration of such salary, within the reach of persons of moderate means, and at the same time to place some sort of check upon the large class of persons who would willingly make use of the services of a public officer—or of any one else—if they could get them for nothing. It was not intended as a basis upon which the salaries of public analysts were to be calculated, or as a typical fee to be paid to a private analyst. But they (the analysts) had themselves to blame for not uniting together to demonstrate these facts in a practical manner to all those whom it might concern. He was most anxious to see some sort of combination among analysts with respect to their common professional interests. Hitherto they had segregated rather than aggregated in reference to these matters, and had accordingly been taken at a disadvantage. Medical officers of health had succeeded in getting a rather better appreciation of the value of their services than had hitherto been obtained by public analysts. The reasons for this were not far to seek, and it was important that the lesson should not be lost upon analysts. It was through the influence that could be exerted by the public analysts, the only officially recognised chemists, that the profession generally might be benefited. It was a mistake to suppose that an extension of official work would injuriously affect private analysts. On the contrary, it appeared to him to be obvious that the effect of such extension would be entirely the other way. In addition, anything which tended to show how great was the importance of properly conducted analytical work to the community, would plainly be beneficial to the profession, and nothing could accomplish this more satisfactorily than the extension of the work of public analysts, who were in contact with the public and with public authorities, and whose results, in consequence of their position, became public property, and were spread over a wide area. But it was essential that they should be united in resisting all attempts to minimise their position or to detract from the value of their work, whether those attempts were made by individuals or by public bodies. He felt that while the extension of the machinery of the Sale of Food and Drugs Act so as to comprise other articles, would be attended with many difficulties, and would meet with much opposition, it was their duty to take steps when the proper time came, as it probably soon would, to secure the analysis by public analysts of certain articles, the unrestricted sale of which was dangerous to the public health. Among these might be mentioned arsenical pigments, articles of clothing, of decoration, and for other purposes, containing arsenic and other poisons, so-called patent medicines, various quack preparations, "disinfectants," falsely so-called, and so on. They would not get everything at once, and more attention was likely to be paid to matters involving injury to health. The subject was an extensive one, but he thought that it would be well if the Society could arrive at a decision on the question of the desirability of extending public analysis. He did not think that their taking unofficial action when the time was ripe for it, would have any of the bad consequences which had been anticipated, or that it would be in any way derogatory to their dignity as professional men. It could not be denied, at any rate, that an extension of public analytical work would be a good thing for the public.

Mr. Cassal stated subsequently that he thought he ought to apologise to the Society for having read a paper at Worcester recently upon "The Extension of Public Analysis," inasmuch as this discussion would have probably led him to modify some of the views he had expressed in that paper, if he had heard it previously. If he had thought that the sum of 10s. 6d. was likely to be introduced as the measure of the value of an analysis in any Act extending public analysis, he would have burnt his Worcester paper without hesitation. He was very anxious to impress upon the Society that no extension should take place unless the 10s. 6d. fee and the 10s. 6d. spirit were got rid of. He adhered to his original view that the only real way of advancing general professional interests was through the influence which could be brought to bear by the official public analysts. The curses of their profession had been, and still were, a want of mutual confidence and a want of united action. If once they got the importance and responsibility of their work more adequately recognised, great benefits would be derived not only by the analysts in this country, but by the profession in every part of the world.

Mr. Davies said that it appeared to him that such an extension was eminently desirable, and he did not think private analysts would view it with the feelings indicated by Mr. Hehner. The direction in which he thought that they might advance was more particularly that of official analysis—such analysis and analytical investigations as were directly of use to the public, and brought to the public analyst by the vestry or the authority by whom he was appointed and for whom he was working. It appeared to him that the question of public risk was one that mainly belonged, in no small degree, to the public analyst, who, after all, worked in the interest of the district, as much as the medical officer of health, or the vestry clerk, or the surveyor, and he did not go outside his duties if he endeavoured to bring about a systematic examination of those articles that were largely used by the public, and brought such examinations into line with his ordinary duties as they at present understood them.

The directions that particularly occurred to him then were the analysis of papers and fabrics that might contain arsenic or other deleterious matters, and disinfectants, and what was now done irregularly by some authorities might be done regularly.

Respecting other matters, such as petroleum, he was not by any means sure that it would be an advantage that that article should come under the public analyst's province, and with regard to the Pharmaceutical Society's functions as to passing it did not appear to him that it would be useful to transfer them to the public analysts. As to whether the Society of Public Analysts should take action he agreed with Mr. Cassal and Mr. Hehner that it would be inadvisable for the society to consider such a matter at present. The time was not a proper one, nor was the Society at all in a position to usefully move in the matter.

Mr. Dyer said that he had had during the day the pleasure of a conversation with Mr. Heisch, who was struck with what seemed to him like a lament on the part of Mr. Allen, that water did not come within the official duties of a public analyst, seeing that it was solely by the anxious persistence of the Society years ago that water was excluded from the Act. As originally drafted the Bill included water, but the Society's then Secretaries, Messrs. Heisch and Wigner, made such representations to those who had charge of the Bill, that the clause was withdrawn; and that was done in the interests of public analysts. As Mr. Heisch pointed out, the public analyst of a district was naturally the man who got the work in his private capacity, and as a rule he got more for it than if his fee was fixed by the Local Board and under the Act of Parliament. With regard to the question of arsenic, there was a bill in existence which had been very carefully drafted, and dealing with it very much on the lines of the Acts of other countries. It had been prepared by the National Health Society, who were awaiting a suitable opportunity to introduce it into Parliament. Care was taken that the official chemist should be the public analyst of the district in which the samples were taken—

all papers, toys, and so on. Also that the Sale of Food Act should be read as part of hat bill as regarded sampling, analysis, and procedure. The bill would be introduced nto Parliament at some time or other, but they had been advised that the present oment was inopportune for dealing with the question. Mr. Allen said that Corpora-ions and Boards of Health suffered largely from deficient strength in the carbolic owders supplied to them. Why should they suffer? They were made up generally f business men, and why did they not submit the powders to the analyst and have them nalysed? An Act of Parliament was not needed for that as was the case with food and ater and milk, or arsenical wall papers where the State might properly step in. But ith Corporations and Boards of Health who send out invitations for tenders and ave samples and guarantees, why should an Act of Parliament be necessary to enable hem to have these things analysed.

Mr. Smetham said he was not a public analyst under the Act, and therefore in tating his opinions he did so from an outside point of view. He had listened to what ad been said that evening and had come to the conclusion that it would be undesirable, nless some provision could be made to raise the fees, to bring into the Act all these utside substances suggested by Mr. Allen. As Mr. Dyer had pointed out, Corporations nd the public at large were pretty well able to take care of themselves as far as the oney value of substances was concerned, but not as regarded the wholesomeness of the ood they consumed. But the Act was brought into existence more particularly for the rotection of the lower classes who were totally unable to guard their own interests, and herefore the State considered it its duty to protect them against the injury to health aused by the fraudulent devices of unscrupulous dealers. The question seemed to resolve tself into what Mr. Allen had called "State Chemists." It was really more a question, f carried to its ultimate limits, of the introduction into England of men who would ndertake the whole of the work that might be required from a chemical point of view. He thought that if such men were appointed it would tend in some instances to bribery nd fraud. More important still the inspection would hamper the various trades and ndustries now in existence. Of course, personally, he had no sympathy whatever with raudulent devices or any adulterations, whether it be soap or what not, but it was cer-ain that the cheaper articles which, as he understood, Mr. Allen would keep a control ver, although adulterated were sold as a rule at their relative market prices and the ublic practically was not cheated. So far as the extension referred only to the protec-ion of health, testing wall papers, etc., then he was certainly in sympathy with Mr. Allen, and the extension he advocates, but when he goes beyond those limits, and seeks o keep a control over ordinary commercial manufactures having no bearing on the ublic health, he thought Mr. Allen was going beyond the province of the public analyst.

Dr. Vieth said that in his opinion it would not be opportune for the Society to ove in the direction suggested by Mr. Allen. Referring to Mr. Cassal's remark, that hey should endeavour to impress the authorities on certain points, one point should be he desirability of magistrates imposing heavy fines where adulteration was proved. Another point was for the authorities to direct the inspector where and when he ought o take samples. He had a particular case in view. They all knew they could pretty asily detect the adulteration of milk with water, but it was most difficult to prove to he satisfaction of the magistrate the abstraction of cream. And as long as samples were aken in the streets that would be so, and if a fine were imposed it might be n the wrong man, some poor fellow who could not protect himself. Samples hould be taken not only in the streets but at railway stations. It was only wo nights ago that forty churns of skim milk were unloaded at one of the ermini, evidently intended for mixing with whole milk. He had it on the authority of an eye witness that a churn of skim milk was divided amongst different churns of whole milk which were not quite full, and, of course, sold as whole milk. If inspectors could take samples at railway stations they would soon put a stop to that sort of thing.

Mr. S. **Harvey** said that he felt disposed to give the paper his cordial support, as Mr. Allen had put his finger upon several weak points. He had been somewhat puzzled by the nice distinction drawn by a previous speaker between the advisability of legislation as regards arsenical papers and fabrics, and the non-interference with such articles as soaps and disinfectants. With reference to the latter, if appeared to him that when they were deficient in antiseptic properties they were at least indirectly injurious to health, because they were assumed to possess specific purifying effects, which did not really exist, and that thus the public would be misled. Referring to soaps, he was an observer some years ago of the effects following the introduction of a softened water supply to a borough previously dependent upon very hard well waters, severe eruptions both upon face and hands became, for a time, common, and were attributed to the caustic lime used in the softening process. His observations induced him to regard the use of impure and cheap scented soaps as the real cause. On the other hand he was personally opposed to over-legislation instancing the regulations at present affecting the sale of poisons. An experience of more that thirty years as a pharmacist had convinced him that the public were better protected, so far as chemists were concerned, prior to such legislation, than at present, when so many poisons were really more accessible than they were at the hands of outside dealers. When accidents happened, the latter were seldom, if ever, censured, while chemists were often exposed to most vexatious persecution, even after compliance with all the regulations enforced by the Act.

Mr. E. G. **Clayton** said he thought it would be a mistake from all points of view for the Society to move for this legislation. It would be bad for all parties—for public analysts as well as private analysts. For instance, if water were included at a legalised fee of (say) a guinea, public analysts would be compelled to make these laborious analyses for that sum, and other analysts would have to do the same.

Mr. **Adams** said that with regard to fabrics, wall-papers and perhaps other things there was great room for improvement so far as the public were concerned, but as to other matters he could not agree with Mr. Allen, as others had been unable to agree with him that evening. As to the first point, whether it was desirable to extend the Act, he agreed so far as the substances mentioned were concerned, but whether it was a proper time, or they were the proper body, he had his doubts. Their being only a small body, it would be difficult to persuade the public that they were not acting a self-interested part. All they could do was to stimulate the authorities through members of Parliament and others. With respect to the time, he thought it was very inopportune. The Public Health Act, generally speaking, was not welcomed by the public at large, nor by the public bodies who had to execute the Act against the inclinations of the general public. In witness of that he might appeal to the fact that where public analysts were appointed by small bodies, the analyst got very little work. This meant that the appointing bodies were against the working of the Act, and they only reflected the feeling of their constituencies. It was only by large bodies, unshackled by trade associations, such as county authorities, that the Act was vigorously worked. For that reason he thought they might assume it was unwelcome and would be unacceptable in a Parliament the members of which would have to look about them for popular favour when they went before their constituents. The proposal must not come through their body, but from members whom they could personally influence, and not at a time when Parliament was on the eve of a change. With regard to water, he did not wish to see t included in the Act, but would very much like the examination of water to be made local business, for he was positive that no one in England knew better than he him-elf did as regarded the water in his neighbourhood (Maidstone), and others no doubt vere in a position rightfully to say the same for themselves, and from that point of iew it would be better that it should be the duty of the public analyst to examine it; ut on the other hand he should be ashamed to see the fee reduced to such a ridiculous nd totally inadequate sum as 10s. 6d. There were several points which had been aised on which he should have liked to say a few words. One was the National Health ssociation, and he hoped they might congratulate themselves that it was still in exist-nce, and at all events, and it is to be hoped, in earnest, in bringing forward the uestion of arsenical poisoning. With regard to disinfectants and soap, he was in ympathy with Mr. Harvey. It was of the greatest importance that the public should e informed of what they used in the dire necessity of an outbreak of fever, although is own experience as medical officer of health led him to place less and less faith in isinfectants.

MR. ALLEN, in reply, said it was not to be expected that everyone would agree with im on all points, but he thought he might safely leave one half of his critics to answer he other half.

He had been misunderstood on one or two points. He never intended to suggest hat drinking water ought to be included in the Act. He simply mentioned that it was ot included at present. Of course there were some instances where, according to the erms of the appointment, the analyst was expected to undertake analyses of water for anitary Authorities in the district for which he acted; but in such cases he believed here was always a special fee allowed. In fact, he was by no means in favour of the 0s. 6d. fee for food analysis, and he thought that one of the very reasons why they hould attempt to get an amended act. If an Act were drafted without their having heir say in it they might depend upon it the fee would not be amended. He quite greed that the trouble and responsibility attached to the making of official analyses nder the Sale of Food and Drugs Act were by no means renumerated by the fee of 0s. 6d., which had undoubtedly been originally introduced for the purpose of giving rivate purchasers the opportunity of having the articles they purchased tested at a oderate charge; it being assumed that the analyst would be recouped out of the salary e received from the authorities appointing him.

Mr. Allen could not agree with those who considered that the present time was nopportune so far as Parliament was concerned. The present Parliament, if it lasted ts natural life, had still three or four sessions before it. The introduction of a new anitary Analysis Act was by no means calculated to bring strong political feelings into onflict, and they might depend upon it that whatever Government introduced a Bill, here would be plenty of cross voting. If the present Government remained in office hey had plenty of time before them to pass a Bill as suggested; on the other hand, f their opponents succeeded to power the objection raised did not apply. In either case, herefore, he thought that they should be prepared. He thought they should appoint a ommittee who should draft a Bill as carefully as possible and then pigeon-hole it. If hen they pressed the matter on members of Parliament with whom they came in ontact and were asked, "What do you gentlemen desire?" they would then only have o produce the Bill and say, "This is what we wish." On the other hand, if they did othing now, the time would come when all would have to be done in a hurry, nd we should find that a Bill was drafted and introduced in which our opinions and nterests were wholly ignored. He did not agree that it was not possible for public nalysts to draft such a Bill. They were undoubtedly the best qualified body of men in the Kingdom to express an opinion on the point, as they best knew the difficulties of the past and the requirements of the future. He was not inclined to insist that the Act proposed should cover all the articles he mentioned. He thought there was a great deal to be said in favour of making the Act one simply relating to sanitary chemistry, and he should be disposed to leave such things as petroleum and mixed fabrics alone. He could not see that it would in any way have an injurious effect upon the interests of chemists who hold no appointment at present under the Sale of Food and Drugs Act. There were undoubtedly a large number of articles which he suggested should be made the duty of the public analyst to examine, which, at present, were not examined at all. If these were brought within the operation of a future Act it would be sure to bring work to the unofficial analysts, who would be consulted by the manufacturers and dealers in the same manner as they are at present, respecting articles subject to inspection. Whether they took any step or not he must remind them that there was sure to be legislation on the subject of poisons during the next Parliamentary Session.

Respecting disinfecting powders, he certainly considered these should be brought within the operation of the Act, and that the vendors should be compelled to state the strength or composition on the label, so that people would not be buying as carbolic powder, preparations containing merely an insignificent amount of the active ingredient. Similarly with sanitary soaps. It ought to be made illegal for a firm to state that their soap contained certain substances having a curative or emollient action, actually naming those constituents, when, as a matter of fact, there was nothing of the kind present.

With reference to the term State Chemists, which he had used in his paper, he might say that the title did not originate with him, but he believed was due to the President. He had simply adopted it as being, in his opinion, a highly suitable title.

Conclusion of the Society's Proceedings.

THE ELECTROLYTIC SEPARATION OF CADMIUM FROM ZINC.

By Edgar F. Smith and Lee K. Frankel.

The separation of these metals has been effected by Yoer (*Bull. Soc. Chim. de Paris*, 34, 18), who employed the acetates for this purpose.

Eliasberg (*Zeitschrift für Analyt. Chemie.*, 24, 548) and Smith and Knerr (*Am. Chem. Journal*, 8, 210) confirmed this observation, at the same time emphasising the fact that the current should be carefully regulated, otherwise unsatisfactory results would be obtained. A solution containing cadmium and zinc as tartrates, together with free tartaric acid, will also yield all its cadmium to a current generating 0·4—0·5 c.c. oxy-hydrogen gas per minute (*Am. Chem. Journal*, 8, 210). We have no record of other salts of these metals having been used for their electrolytic separation, and therefore taken occasion to learn what might be the result with solutions of the double cyanides, such as we employed in separating mercury and copper (*Journal Franklin Institute*, 127, 386, and *Am. Chem. Journal*, 11, 264).

Beilstein and Jawein (*Ber. d. d. chem. Gesellschaft*, 12, 446, 762) have shown that both cadmium and zinc can be completely precipitated from a cyanide solution, but have not given the current strength employed by them in their experiments, nor did they attempt the separation of these metals from each other.

As our experience with mercury and copper clearly indicates, it is frequently possible by close attention to the current, to effect electrolytic separations, which ordinarily mpossible. With this fact clearly before us, our first work with the metals now consideration was to ascertain how feeble a current would suffice for the complete tion of each metal, when alone in a cyanide solution, in the presence of an excess alkaline cyanide. We very soon discovered that the cadmium separated readily ith a much weaker current than was necessary for the deposition of the zinc. tter will, however, separate from a cyanide solution, even under the influence of a current, but not until the excess of cyanide has been completely decomposed. the quantity of cyanide used by us, and with a current of the strength indicated this complete decomposition of the alkaline cyanide is not likely to occur in a r period than forty-eight hours. Hence, it follows, that as the quantity of cadused in our experiments is entirely precipitated in a little more than eighteen the complete separation of cadmium from zinc is thoroughly feasible by this d.

ll our experiments were conducted in the cold, and care was taken in each case to e the deposited metal for zinc, and the residual solution for cadmium. The con under which our work was carried on and the results obtained are these—

	Cadmium found.	Metallic Zinc present.	Difference in per cent. of Cadmium.	KCN in Grammes.	Total Dilution.	Current in c.c. of O-H Gas.	Time.
			Per Cent.				
7	·1813	..	– ·22	4·5 grammes in each separation.	200 c.c.	0·3 c.c. in each experiment.	18-23 hours.
	·1818	..	+ ·05				
	·1815	..	– ·11				
	·1822	..	+ ·22				
	·1812	·2000	– ·27				
	·1818	·2000	+ ·05				
	·1828	·2000	+ ·60				
26	·2422	..	– ·16				
	·2432	..	+ ·24				
	·2426	..	..				
	·2420	·2000	– ·24				
	·2435	·2000	+ ·30				
	·2429	·2000	+ ·12				
	·2426	·2000	..				
	·2433	·2000	+ ·28				
	·2434	·2000	+ ·32				

The cadmium deposit was light grey in colour and crystalline in structure. It ashed with hot water and dried upon a warm iron plate.

Chemical Laboratory of the Univ. of Pa., Philadelphia, Pa.

SOME CAUSES OF ERROR IN BLANK ANALYSES.

By J. B. Mackintosh.*

In making chemical investigations, it is quite usual to run a blank analysis, in order to apply a correction to our results for the impurities contained in the reagents and for those entering into solution from the apparatus used. As a rule however, it seems to be entirely overlooked that the conditions of the actual analysis are often quite different from those obtaining in the case of the blank. For instance, let us assume the case of a limestone which has been dissolved in just sufficient hydrochloric acid to form a neutral solution of calcium chloride; at this stage our blank analysis will be a strongly acid solution. If we now add to both enough ammonia to neutralise the amount of hydrochloric acid previously employed, we will have in our actual analysis a strongly ammoniacal solution, while in our blank there will be a neutral solution of ammonium chloride, and similar results will take place on the addition of every reagent. In such a case our blank analysis will give us only approximately correct results, for though any impurities existing in the chemicals will be detected, yet the impurities which are dissolved from our beakers by the solutions, will be quite different in the case quoted, and the errors due to this last source may far exceed those due to impurities contained in the chemicals used.

The particular case of blank analysis to which I desire to call attention, serves to illustrate the serious error into which chemists may be led by faith in the infallibility of books of reference, whose statements, copied from one another, gain in authority through force of iteration, even though, as sometimes is the case, the original statement be erroneous.

Several years ago I had occasion to analyse a large number of samples of metallic copper and alloys of copper, and for the determination of arsenic I decided to use the method of distillation with ferric chloride and hydrochloric acid recommended in Crooke's Select Methods.† In that work the statement is made that the ferric chloride employed may be freed from all traces of arsenic by one or two evaporations to dryness with hydrochloric acid, whereby the arsenic will all be volatilised as chloride. In following these directions I collected the distillates, thus making a blank analysis in all respects similar to the actual analyses performed later. When the distillates were tested for arsenic they were found to be perfectly free from that element, from whence the deduction was drawn that the reagents employed were pure. If the statement mentioned above was correct, then this deduction was justified by the facts observed. I noticed, however, that all the samples of copper I had, no matter of what quality, invariably caused the appearance of large quantities of arsenic in the distillates, and on repeating the experiment with the substitution of some charcoal for the copper, I likewise obtained arsenic in the distillate, and since the charcoal could not be suspected of being the source of the arsenic found, it was definitely traced to the ferric chloride, which by a blank analysis would have been pronounced pure. In this case the omission of the copper in the blank alysis was also the omission of a necessary reagent, without which or some substitute, e reaction would not take place. In fact, it is not until the arsenic is reduced to senious chloride that it will distil off, any arsenic in the higher form of oxidation not ing volatile under these circumstances.

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* *Journal of Analytical Chemistry.*

† First Edition, p. 266; Second Edition, p. 431.

Here is a striking instance of the unreliability of blank analyses, unless all the cumstances be thoroughly comprehended and taken into account, and other instances similar nature could readily be adduced.

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Use of Oxygen in Quantitative Analysis. W. Minor. (*Zeitschr. f. angew hemie*, No. 23, 1889.—It seems strange so little use is made of oxygen in chnical laboratories. Take for instance wine analyses, where the getting of a proper h, without volatilising chlorides, is a difficult operation. If, however, the extract is ated in a current of oxygen, the ash is got ready in a few minutes. Another instance furnished by cane-sugar and allied bodies. Although this is generally first moistened th sulphuric acid and the weight of the ash multiplied by ·9, still the treatment in ygen will dispense with the use of the acid. In the estimation of carbon in pig-iron, e use of oxygen is very advantageous. The iron is placed in solution of cupric loride, which gradually dissolves the iron, leaving an impure carbon. This is collected an asbestos filter dried in a platinum crucible at 120° C. and weighed. The carbon is en burnt in oxygen and found from the loss in weight. Oxygen will also be found vantageous in the estimation of sulphur or sulphates. The well-washed precipitate of rium sulphate is put moist in a platinum crucible, and after charring ignited in a rrent of oxygen. This will prevent all chance of reduction, and the operation is ne in a few minutes.

L. de K.

Borgman's Process for the Estimation of the Acids in Wine. R. Gans. *eitschr. f. angew Chemie*, No. 23, 1889).—This process, consisting in treating the ne extract with alcohol, to precipitate the cream of tartar and dissolve out the ee acids, has been found by the author to yield uncertain results. As the results of s experiments he concludes :—1. The estimation of the cream of tartar is influenced the amount of sugar. If much sugar is present some of the cream of tartar does t separate out, and some free tartaric acid remains undissolved. 2. The estimation of e free tartaric acid (by means of potash) is also inaccurate, as especially in absence of fficient sugar nearly the whole of the acid potassic malate also separates out. In fact, e process can only be trusted to give approximate results in the case of very sweet ines.

L. de K.

Detection of Small Quantities of Nitrous Acid. G. Lunge. (*Zeitschr. f. angew hemie*, No. 23, 1889.)—A well-known disadvantage of the otherwise so excellent agent of Griess for nitrous acid, is the circumstance of the solution, even of the hitest looking (*α*) naphtylamin, getting dark and therefore losing in sensitiveness. hen the solution to be tested is very dilute (1-1,000000000), the test works so slow, that one cannot be certain whether the nitrous acid has not been absorbed from the atmosphere. Even after warming, which facilitates the reaction, the test may not show for 15 or 20 minutes. Jlosvay shows that by using acetic instead of sulphuric or hydrochloric acid the reaction takes far less time, and the colour is also intensified. He also states that the darkening of the reagent may be prevented by boiling some solid naphtylamin with water, and only using the fluid which has been poured off from the residue. In fact, he gives the following recipe:—1. Dissolve ·5 gramme of sulphanilic acid in 150 c.c. of dilute acetic acid. 2. Boil ·1 gramme of solid (*a*) naphtylamin with 20 c.c. of water, pour off from the blueish deposit, and mix with 180 c.c. of dilute acetic acid. The liquid to be examined (about 20 c.c.) is mixed with a few c.c. of the sulphanilic acid, warmed till about 80° C., and then mixed with the solution of the naphtylamin. Even when the solution contains only one thousand of a millionth part of nitrous acid, the red colour will appear after one minute. If the proportion of the nitrous acid is larger, say 1 in 1000, only a yellow colour will appear, unless a large quantity of naphtylamin is added. The author on the whole agrees with Jlosvay, only thinks he can still further improve it. The test for nitrous acid is just as delicate and quick, if two reagents are mixed and kept in a well-stoppered bottle. Air must be as much as possible excluded, but it is not necessary to keep the reagents in the dark. By this small modification we get the great advantage of using *one* solution only. Then if the two solutions are kept separate, one can never tell whether perhaps one of them has absorbed a little nitrous acid from the air. If, however, the mixed solution should absorb any nitrous fume, it would at once betray this by the reddish colour. If a solution should have got coloured, it is often possible to restore it to its original condition by shaking it with zinc dust and filtering. L. DE K.

Estimation of Iron and Alumina in Phosphates. E. Glaser. (*Zeitschr. f. angew Chemie*, No. 22, 1889.)—The process for the estimation of iron and alumina in phosphates, as now practised, is not applicable to any variety of phosphate, and therefore not very useful. Its shortcomings have shown themselves more particularly in the last few years, when many samples of Belgian and similar phosphates are on the market. The results are somewhat dependent on the amount of acetic acid used, then aluminic phosphate is somewhat soluble in excess. This conventional process is as follows: The precipitate insoluble in acetic acid is filtered off, washed with cold water, and then dissolved off the filter with hydrochloric acid. After adding a little microcosmic salt the liquid is rendered alkaline with ammonia and then mixed with acetic acid. The precipitate is washed, dried, and ignited, and half of its weight is called iron and alumina. No wonder analysts differ in their results from 1 to 3 per cent. Not only, however, is the error solely due to the slight solubility in acetic acid, but also to the fact that in the original sample, the iron is not always in combination with phosphoric acid. When such a solution is precipitated with ammonia, the lime and magnesia are precipitated as phosphates, which is also the case with the iron and alumina, if there is a sufficiency of phosphoric acid. This, however, is not always so, and then there forms a precipitate of ferric and aluminic hydrate, which redissolves in excess of acetic acid and so get lost.

in the ferric acetate has a solvent action on ferric phosphate. If too little d is used the precipitate may contain lime and the result is equally faulty.

te by abstractor: Iron and alumina phosphates will not properly come down nonia and acetic acid if the phosphate contains organic matter, which is very case. This being absent, all iron and alumina will certainly come down, cases where there is a want of phosphoric acid).

author now tried to find a process which will dispense with the acetic acid, and by the use of alcohol as follows: 5 grammes of phosphate are dissolved as 25 c.c. of nitric acid of 1·2 sp. gr., and 12·5 c.c. of hydrochloric acid, and diluted c.c. 100 c.c. of filtrate (= 1 grm. sample) are put into a 250 c.c. measure and th 25 c.c. of strong sulphuric acid. After standing for about five minutes and l shaking, 100 c.c. of alcohol are added, and the whole well shaken. After lcohol is added up to the mark, but as there is always a little contraction it will necessary to later on add some more spirit. After at least standing for half he liquid is filtered, and 100 c.c. (= ·4 grm. sample) are evaporated in a dish till alcohol is expelled. The acid fluid is rinsed into a beaker with c.c. of water, and heated to boiling. After cooling a little ammonia is added e reaction, and if necessary the excess of ammonia is expelled by boiling. The e is now collected, well washed, dried, and ignited. It consists of pure ferric and pure aluminic phosphate. The iron phosphate can be determined by a ic estimation, say with permanganate.

test analyses given are very satisfactory, more so than the analyses performed nventional process. L. de K.

ECTION OF GROUND-NUT OIL IN COD-LIVER OIL. V. BISHOP. *Journ. de ph*, , xx. *p*. 302.—Ground-nut oil is frequently added to cod-liver oil with a double ., to give to the Cod-liver oil a more pleasant taste and to increase the profits of or. It is therefore to our great interest to be able to detect it, and seeing that it ossess characteristic properties, as does, for instance, oil of sesame, its detection is ate and uncertain. Nevertheless, having studied the question the author believes uite possible to arrive at a satisfactory solution. The colouration furnished by tably by sulphuric acid, the estimation of the iodine, etc., do not always give ications. The density, the heating power of the sulphuric acid, the absorption ne are three modes of analysis capable of giving much more exact results, on f the notable differences which exist in these respects between the two oils.

eply, then, the author works in the following manner:—

The density is taken by the areothermic balance with the usual precautions.

The action of sulphuric acid on cod-liver oil and even on ground-nut oil being getic, and producing a strong disengagement of sulphurous acid, it is indis- to mix them with a known quantity of a retarding oil, the heating power of at once low and ascertained. With this object the author employs heavy oil, which, as is well known, possesses a very low point of heating power, huric acid, and such oil he mixes with the cod-liver-oil in equal proportions.

In applying the bromine absorption method, the author employs M. s process, by which the oil is saponified and the fatty acids liberated in the nner. 1 c.c. of these acids having been dissolved in 20 c.c. of carbon bisul- e solution is treated with a fixed excess of a standardised solution of bromine.

After standing the usual time, the unabsorbed bromine is estimated by a standard solution of soda, strongly coloured with cosine, On applying these methods to standard samples, the author has obtained the following results :—

	Density to 15°.	Heating power with sulphuric acid 10 gr. oil. 10 gr. of mineral oil. 20 gr. of sulph. acid.	True heating power.	Absortion of bromine.
White cod-liver-oil ..	0·9265	67°	106°	0.732
Light " " ..	0.9257	65°	102°	"
Brown " " ..	0.9264	65°.25	102°.5	"
Ground-nut oil ..	0.917	48°	66°	0.534
Mixture, 21 % ground-nut " 80 % white oil	0.9243	62°.5	97°	"
Mineral oil ..	0.9243	14°	97°	"

M. S. A. M.

REVIEWS.

An Introduction to Chemical Science. By R. P. Williams, A.M., Instructor in Chemistry, English High School, Boston, U.S.A. Edited and Revised by B. P. Lascelles, M.A., F.C.S., Assistant Master and Librarian, Harrow School. London: Ginn and Company.

This is decidedly the best practical book we have yet seen for use in schools. It is essentially an elementary book, and is not written with the view of examination, but is formed on a specially interesting plan leading the learner through a series of experiments which convey to him a very fair idea of the chemistry of common things. Commencing with weights and measures and passing through a short account of what chemistry is, and of atoms, molecules, etc., the authors go first through the non-metals, then take up some of the chief industrial metals, and finally indulge in a little dip into organic chemistry, mentioning such matters as gas-making, fermentation, soap manufacture, etc. A boy working through the book in a school laboratory would acquire exactly what a schoolboy should, viz., a good groundwork enabling him to have ideas as to the composition of the articles he sees around him, and of the chief industrial chemical processes. It is true that he would not be specifically prepared for any particular examination, but he certainly would have much broader views than boys that are so taught, and we should say that he would pass the College of Preceptors or any such preliminary very easily in the subject of chemistry. Every schoolmaster who has a laboratory should get a copy of this book and let his boys use it if he wants them to be really interested in the study of chemistry. It is printed in good, bold, readable type, and extends to 223 pages, 8vo. size, at the moderate price of 3s. 6d.

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the Analyst.

Sir,—In reference to the communication from Mr. Percy Smith in the November number of The Analyst, we desire to say that, while it might appear to any one reading the review of our "Water Analysis" in the preceding number, that we had adopted, without credit, the explicit descriptions given by Mr. Smith, yet, as a matter of fact, due credit is given in the preface to the work. The reviewer, by inadvertence, omitted to note this fact. Analysts are certainly under obligations to Mr. Smith for having demonstrated the trustworthiness of these methods, and furnished clear and practical descriptions for their employment.—Yours,

Henry Leffmann.
William Beam.

715, Walnut Street, Philadelphia,
November 22nd, 1889.

THE ANALYST.

FEBRUARY, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual Meeting of the Society was held on the 8th ult., at Burlington House, the President, Mr. Allen, in the chair.

The minutes of the previous meeting were read and confirmed.

The Treasurer, Mr. Heaton, read the Auditor's report, showing a balance of £161 in the bank, besides the £100 already invested in Consols.

Dr. Muter thereupon moved, and Mr. Harvey seconded, that the accounts be passed as read, and that a further sum of £100 be invested in Consols.

This was carried unanimously, and the Auditor's report will be circulated amongst the members as usual.

The President then delivered his annual address as follows:—

GENTLEMEN,—My first duty is to thank you most cordially for the support and consideration shown me during my year of office as your President, and I beg you to believe that it has always been to me an honour to be reckoned one of, and far more for the nonce to be the representative, of a body of men who deserve to rank as high as any among the day-labourers in the field of science; for may we not justly claim, without presumption, that during the period of the existence of this Society our labour has been as toilsome, as responsible, and as honourable as any to be found.

For toil I see no prospect of abatement. Year by year, as knowledge ripens, the processes we employ become more and more exacting of skill and attention, without, by-the-bye, any increase of remuneration.

For responsibility, it is a question if any calling within the domain of applied science throws more burden upon its followers than that of the public analyst, from whom it is required in the exercise of his never too popular function, to explore regions beset by enemies and pitfalls, without a chart, without a guide, with nothing, in fact, but a sense of duty to keep him to his task.

It is, therefore, all the more *honourable* to public analysts that these difficulties have been so courageously met, and, in large measure, overcome; the credit of conquest falling chiefly to past or present members of this Society.

In pursuit of these ends we have during the past year held eight general and an equal number of Council meetings. At these meetings twenty-three papers have been read; the attendances have been above the average, and the discussions well sustained.

The following is a list of the contributions:—

Papers in 1889.

Date.		Author.
Jan. 9.	"Remarks on a Recent Case of Alleged Injurious Pollution of the River Medway by the Sewage of Maidstone"	Dr. Dupré
"	"On the Analysis of Pepper and the occurrence of Piperidine therein"	Dr. W. Johnstone
"	"On the Westphal Balance"	W. F. K. Stock
Feb. 13.	"On the Composition of Milk and Milk Products"	Dr. Vieth
"	"An Improved Method for the Analysis of Fats"	Dr. Muter and L. de Koningh
"	"Note on Phytosterin"	O. Hehner
"	"On Density"	H. D. Richmond
March 13.	"Notes on Lactose"	E. W T. Jones
"	"Colouring Matter (Arsenical) used for Colouring Candles"	W. F. Lowe
"	"The Methods for Determining Fat in Milk"	Dr. Vieth
April 10.	"Contributions to the Study of the Action of Water on Lead Pipes"	A. H. Allen
"	"Iodine Absorption of Butter Fat"	Rowland Williams
"	"'Pure' Chemicals"	Bertram Blount
"	"Some Experiments on the Soap Test	Dr. E. Waller
May 8.	"On the Discrimination of the Various Species of Saccharomycetes"	Dr. W. J. Sykes
"	"Fat Extraction from Milk Solids"	H. D. Richmond
June 12.	"On Condensed Milk"	H. Faber
"	"On the Quantity of Volatile Acids present in Butter Fat"	Dr. Vieth
"	"On the Amount of Volatile Acids in Butter Substitutes"	O. Hehner
Nov. 13.	"A New Form of Air-Bath"	M. A. Adams
"	"The Possible Future Extension of the Duties of Public Analysts"	A. H. Allen
"	"On some Applications of Centrifugal Action in Laboratory Work"	Dr. H. Leffmann and W. Bean
Dec. 11.	"Adjourned Discussion on Mr. Allen's Paper on the Duties of Public Analysts"	

Milk, butter, and fats continue to attract a large share of attention, nine out of the twenty-three papers being devoted to one or the other of these subjects. The contribution of Dr. Muter and Mr. L. de Koningh in February, on "An Improved Method for the Analysis of Fats," gave hope that the year might bring forth some welcome addition to the pressing want of better knowledge of the constitution of, and a means of discrimination between fats, this paper, confessedly an introduction only to a consideration of this important subject, was to be followed by other contributions from the same gentlemen on the application of their process to the analysis of solid fats used for food. I am sure we all regret that as yet Dr. Muter and his co-worker have not been able to redeem a promise which seemed to open out the hope of a new channel for the investigation of the proximate constitution of fats; and the more so, seeing that the work of the Committee appointed by the Society at the end of last year to investigate the subject has been entirely barren of results. But it must not, on this account, be considered that our knowledge concerning the analysis of fats has stood still; far from it. The rays of light that have already been shed upon the subject are being gathered more and more to a focus by the labour of a host of experimenters ; and in this manner the work, being consolidated into a body of experience of the methods of analysis already to hand, is a result in itself to be counted an important gain, and which, it is to be hoped, may form the basis for the acquisition of additional knowledge in the future.

The papers by Mr. Hehner, Mr. A. Williams, and Dr. Vieth have been full of valuable material of this description.

A paper by Mr. Allen, at the November meeting, on "The possible future extension of the duties of public analysts," was full of interest, and gave rise to a long and animated discussion, the mention of which affords me the opportunity for expressing a hope that when fresh legislation is about to take place the opportunity may be secured for pressing upon the legislature the great need of providing some system for the fixing of officially authorised limits, that shall be fair to traders, fair to the public, and binding on the part of analysts and the official referee. Something of this sort becomes more and more urgently necessary as accumulated experience tends to widen the range of natural variation that we find to occur in the constitution of some natural products, milk and butter to wit, this range of variation being so great as to cover, to all intents and purposes, habitual adulteration to a very large extent.

As things now stand, the trader, the public, the analyst, and the referee, all, indeed, but the adulterator, suffer for want of some system of this sort. The honest trader knows that dishonesty, so long as it is kept within scientific limits, will enable a neighbour to undersell him with impunity ; the public, in proportion as this is possible, are unprotected. The analyst, in the dilemma between duty and self-interest, treads a path often as uncertain as shifting sand, and the official referee, however competent, however just, must at times be sorely puzzled where to draw a line that shall be just between all parties.

It may fairly be argued that in some cases, owing to this natural variation of composition, it is difficult to fix a limit; just so, that is only too true, and is the strongest possible argument in favour of a limit being fixed. The difficulty has to be faced in any case, and in the present state of affairs this difficulty has to be dealt with over and over again, instead of once for all, by each and every analyst up and down the country wherever the difficulty arises, according to his own light, as best he may, and thereby the burden of the difficulty is enormously extended, and in practice the duty is discharged in an uncertain, and not always in the most satisfactory manner.

Time was when the course I am advocating was impracticable. We need only look as far back as the time of the inauguration of this Society to find universal ignorance regarding the natural variations of composition of such articles as milk, butter, and lard. At that time it would have been unwise, if not impossible, to fix any limits, but nowadays such ground for objection no longer exists, by the vast accumulation of knowledge appertaining to these matters arising out of the work of public analysts, and in large measure contributed by the past and present members of this Society, it would be a comparatively safe and easy matter to determine what ought to be considered just and reasonable limits for those and other articles.

So far as the present argument is concerned, it is not a question who shall fix the limits, nor whether they shall be high or low, permanent or provisional. As practical men, as working analysts, our chief concern is to have authoritative limits of one kind or another, plain, open, and above board, to work to. If I am not mistaken, we shall all be of one mind as to this, and I think you will also agree with me that it would not be unreasonable that for some things such limits need only be provisional, and subject to review as from time to time the development of the principles and practice of analysis make alterations or modifications desirable ; but as this is matter for the future, and, very likely, not the very near future, a discussion of the details may well be left until there is more immediate prospect for useful action. Remember it was Mr. Allen's paper that brought the subject of future legislation and the particular interest of our Society in connection therewith to the front, and we may now pass to other matters having more present importance for us.

I have to record that our roll-call comprises the following:

Honorary Members	9
Ordinary Members	163
Associates	27
Total	199

During the year we have been deprived by death of perhaps our most illustrious, and certainly most venerable member, Monsieur Michael E. Chevreul, F.R.S., at the surprising age of one hundred and two years.

Two other members are lost to us by the same cause, viz., Mr. Herbert L. Buckeridge, F.C.S., of London, and Mr. J. W. Montgomery, F.C.S., of Whitehaven, Cumberland, and there have likewise been three resignations.

On the other hand, our ranks have been recruited by the election of seven members and three new associates, so that numerically we have a balance of four to the good.

By the resignation of Mr. Frederick Low, our Honorary Solicitor, caused by his approaching call to the Bar, our Society has sustained a distinct loss. Mr. Low is a very able lawyer and excellent advocate, and during the early part of the year rendered successful service in the interest of one of our most respected members, and through him the Society as a whole, in a case involving an important matter of principle respecting the duties and tenure of office of a public analyst.

I am fortunately in a position also to record that, as regards our finances, we have an increasing balance to our credit of some £63; in fact, in all material concerns, we are in a prosperous condition.

The President moved a vote of thanks to the President and Council of the Chemical Society for the use of the rooms during the past year and it was carried unanimously.

The following paper was read and discussed:—"A Rapid Method of Estimating Quinine in Medicine." By Dr. Seaton and Mr. Richmond.

Mr. Allen proposed, Dr. Muter seconded, and Mr. Cassal supported a vote of thanks to the President for his address, and it was carried unanimously.

The Scrutineers, having opened the ballot papers, reported that the following were elected as President and Council for the current year:—

President.—M. A. Adams, F.R.C.S.

Vice-Presidents (who have filled the office of President).—A. H. Allen; A. Dupré, Ph.D., F.R.S.; C. Heisch; Alfred Hill, M.D.; J. Muter, Ph.D., M.A., F.R.S.E.; (Who have not filled the office of President).—Sir Chas. Cameron, M.D.; T. Stevenson, M.D., F.R.C.P.; W. J. Sykes, M.D.

Treasurer.—C. W. Heaton.

Hon. Secretaries.—Bernard Dyer, B.Sc.; Otto Hehner.

Other Members of Council.—S. Harvey; G. H. Ogston; Boverton Redwood, F.R.S.E.; E. Seaton, M.D., F.R.C.P.; E. W. Voelcker.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are C. E. Cassal; R. H. Davies; W. Fox; R. H. Harland; W. Sedgwick Saunders, M.D.; P. Vieth, Ph.D.; and C. R. Alder Wright, D.Sc., F.R.S.

The following gentlemen were also elected as Members of the Society:—W. Newton, Ph.D., London; G. H. Masson, Trinidad.

The next meeting of the Society will be held at Burlington House, on Wednesday, he 12th inst., at 8 o'clock.

NOTE ON SOME APPLICATIONS OF CENTRIFUGAL ACTION TO LABORATORY WORK.

By Henry Leffman and William Beam.

(Read at Meeting, November 1889.)

THE efficiency of centrifugal machinery in various separations has been long known, and inds many applications on the large scale. Special uses in analytical work for examination of milk and the drying of precipitates have recently been brought to notice. In connection with experiments on a small machine, modelled after the lactocrite, and ntended for use by creameries, we had occasion to note that the principle could be advantageously extended to many laboratory manipulations. The first experiment was made on a sample of slightly turbid urine, with a view of obtaining the suspended matter for microscopical examination. This requires several hours usually, by the ordinary method of subsidence, but we found that a rotation of about one minute, at a speed of 16 revolutions per second, produced a clear liquid, with the entire suspended matter closely packed at the bottom of the tube, this condition permitting the decanting of almost the whole of the liquid without disturbing the deposit. Similar separations were obtained with recently precipitated barium sulphate, calcium oxalate, and Prussian blue. A sample of powdered cocoa, adulterated with starch, on being diffused through water and placed in the tube, showed, after a short rotation, a marked separation of the starch, which appeared as a white stratum in the mass of settled material. We have also found that in extraction by immiscible solvents the separation of the solvent can be rapidly and thoroughly attained. Thus the obstinate emulsion which chloroform sometimes produces with aqueous liquids is at once separated.

The essential feature of the machine we used is that the tube carriers are connected by a hinge to a vertical, revolving rod. This permits the tube to hang upright when at rest, taking a horizontal position only when in rapid rotation. It is, therefore, not necessary to close the tubes with a stopper.

The machine used in the above experiments carried eight narrow tubes, about welve centimetres in length, and holding but six c.c. These being too small for regular aboratory work, we are having a machine built to carry tubes of much larger size, and we hope soon to describe it in detail, and many applications of it.

(Conclusion of the Society's Proceedings.)

THE ANALYSIS OF BAKING POWDER.

By C. A. Crampton, Assistant Chemist U.S. Department of Agriculture.*

Baking-powders may be conveniently classified according to the nature of the acid constituent they contain. Three principal kinds may be recognized as follows:

(1) Tartrate powders, in which the acid constituent is tartaric acid in some form.

(2) Phosphate powders, in which the acid constituent is phosphoric acid.

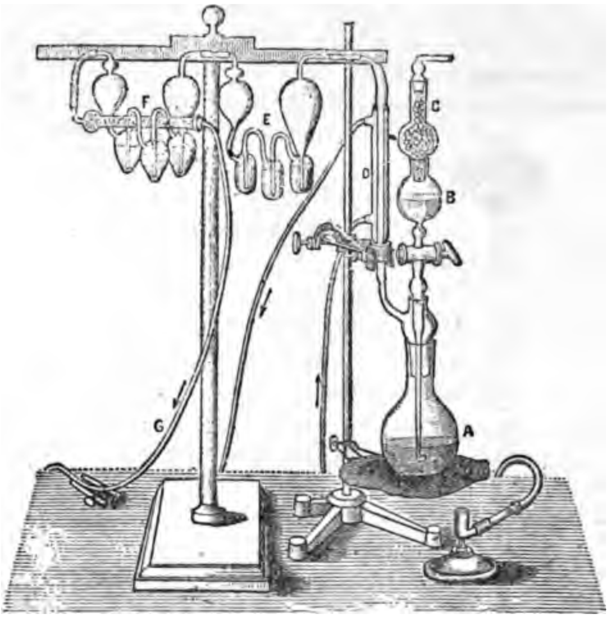
(3) Alum powders, in which the acid constituent is furnished by the sulphuric acid contained in some form of alum salt.

All powders sold at present will come under some one of these heads, although there are many powders which are mixtures of at least two different classes.

The qualitative examination and assignment of a sample to one of the above classes presents no special difficulties. The microscope settles the filling used, but this is usually unimportant. A determination of the alkalinity of the watery solution of the powder is useful as showing whether any great excess of alkali has been used.

Estimation of Carbonic Acid.

This is one of the most important estimations, as it determines the strength of the powder. It was made by absorption with soda lime, and a form of apparatus was used that has served for some time in this laboratory for the determination of carbonic acid.



This apparatus has recently been somewhat modified and greatly improved in compactness by Mr. A. E. Knorr. It is shown in the accompanying figure. The following is Mr. Knorr's description :—

The apparatus proper, as represented by the cut, consists of a flask A in which the carbon dioxide is set free. A condenser D is ground into the neck of this flask and condenses the steam formed when the liquid in A is boiled in order to secure complete expulsion of the gas. The reservoir B contains

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[*] Bulletin of Department, No. 13.

the acid required for the operation, and has a soda-lime guard C ground into it to retain the carbonic acid of the air, a constant current of which is aspirated through G during the whole operation. The stem of the reservoir is ground into the condenser, or it may be conveniently blown into one piece with it. The carbonic acid is dried in E and finally absorbed in the weighed potash bulb F.

Two determinations of carbonic acid were made on each sample—one by the addition of acid to determine the total amount of carbonate present, and one by the addition of water only. The per cent. of carbonic acid found in the latter estimation may properly be termed the *available* amount present in the powder, as it is the quantity which would be actually liberated by the acid ingredient of the powder when it is used in baking, and therefore represents the actual *value* of the powder for aerating purposes, so far as the *evolution* of gas is concerned.

For the determination of the *total* CO_2 the procedure was as follows: Place in a short glass tube, the weight of which is known, about 1 to 2 grams. of the sample, and weigh the whole as quickly as possible, the amount taken being obtained by difference. The tube and contents are gently dropped into the generating flask of the apparatus, which must be perfectly dry. The flask is closed with the stopper carrying the tube connecting with the absorption apparatus, and also the funnel tube, which has been previously provided with 10 cubic centimeters of dilute sulphuric acid for the liberation of the gas. When all parts of the apparatus are connected, and seem to be tight, the stopper of the funnel tube is opened, and the acid allowed to run slowly into the flask. The generation of the gas should be made as gradual as possible; by running in a small quantity of the acid at first and tilting the flask slightly this can readily be accomplished; after the greater part of the sample has been acted upon by the acid and before the latter has all been added, a lamp is placed under it, and the contents gradually heated to boiling, gentle aspiration being made at the same time. The operation is finally finished by the funnel tube being opened, and air, free from CO_2, drawn through it and through the apparatus, the contents of the flask at the same time being kept at ebullition. This is continued for fifteen minutes, when the absorption tube is removed from the apparatus, allowed to cool, and weighed. Its increase in weight gives the amount of CO_2 absorbed. The determination of the *available* CO_2 was conducted in a similar manner, with the substitution of pure boiled water in the funnel tube instead of acid. After the sample has all been acted on, the contents of the flask are just brought to a boil, then the lamp is removed and air is drawn through the liquid for exactly fifteen minutes. The conditions were made as nearly alike as possible for each sample in this estimation, for, different results can readily be obtained by varying them. The above conditions are believed to be as close an approximation to those actually obtaining in the use of the powder as can be arrived at in an ordinary chemical analysis. Prolonged boiling of the liquid residue is inadvisable, for in case the ingredients in the powder are not accurately proportioned, and a considerable excess of bicarbonate is present, long boiling will liberate gas from it after the acid ingredient has all been neutralized, and thus a high result is obtained from a poorly-made sample, while in one where the bicarbonate is not greatly in excess of the proper amount, the above procedure will readily give the full amount available.*

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*In some experiments made to determine the amount of carbonic acid driven off from bicarbonate of soda on heating its water solution, the following results were obtained: (1) Just brought to a boil under the same conditions as in the determinations made above, 6.99 per cent. of the weight of the bicarbonate was obtained; (2) Boiled 15 minutes, 16.17 per cent. was obtained; and, (3) boiled in 1¼ hours, 20.70 per cent., or *about the full* quantity of acid carbonate.

ESTIMATION OF STARCH.

This estimation was made by the well-known method of conversion by heating with acid and the determination of the copper oxide reducing power of the resultant solution. While by no means satisfactory, this is probably the best method we have at present for starch estimation. No difficulty was found in its application to all classes of baking-powders, the other ingredients offering no obstacle to its proper performance. To insure agreeing results it is very essential to conduct the conversion under precisely the same conditions in all cases.

The following is the detailed procedure:

From 2 to 5 grams. are weighed out and transferred to an Erlenmeyer flask; to it are added about 150 to 200 cubic centimeters of a solution of hydrochloric acid which has a strength of 4 per cent. of the acid gas. The flask is provided with a cork, perforated for the passage of a condensing tube about 1 meter in length. The conversion is accomplished by gently boiling the acid liquid for four hours, after which the flask and contents are cooled, neutralized by the addition of sodium hydrate, made up to a definite volume and the copper oxide reducing power determined. The latter operation is best carried out by the method used in this laboratory, in which asbestos-tipped filtering tubes are used for the end reaction.[*] The reducing power being calculated as dextrose; 10 parts equal to 9 parts of anhydrous starch.

Professor Weber used a rough method for the direct estimation of starch in his samples, which he describes as follows:[†]

> One gram. was weighed off, transferred to a small beaker, covered with water, allowed to stand until action had ceased, filtered and washed, residue spurted by means of a wash-bottle into a flat-bottomed platinum dish, allowed to settle, the supernatant water removed as far as possible by means of a pipette, the remainder of water evaporated, the residue dried at 100° C. and weighed. The residue was then incinerated and the amount of ash deducted from above weight. In case of alum powders the ash remaining after ignition was Al_2O_3, which was contained in the residue dried at 100° C. as $Al_2(OH)_6$; consequently the Al_2O_3 was calculated as $Al_2(OH)_6$ before deducting.

This method was carried out upon the entire series of samples examined here. In many cases it gave results agreeing quite closely with those obtained by the direct estimation, but in some samples the results were entirely too high. It is not applicable to alum powders even with the correction made above. For a rough method it answers fairly well and it is quite easy of execution.

ESTIMATION OF PHOSPHORIC ACID.

This determination was made in the same manner as in fertilizers, as prescribed by the Association of Official Agricultural Chemists at their last meeting, as follows:[‡]

Weigh out 2 grams. of the sample, ignite carefully in a muffle, and treat with 30 cubic centimeters concentrated nitric acid.

Boil gently until all phosphates are dissolved and all organic matter destroyed; dilute to 200 cubic centimeters; mix and pass through a dry filter; take 50 cubic centimeters of filtrate; neutralize with ammonia. To the hot solutions for every decigram. of P_2O_5 that is present add 50 cubic centimeters of molybdic solution. Digest at about 65° C. for one hour, filter, and wash with water or ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the ecipitate on the filter with ammonia and hot water and wash into a beaker to a bulk not more than 100 cubic centimeters. Nearly neutralize with hydrochloric acid, cool, d add magnesia mixture from a burette; add slowly (one drop per second), stirring gorously. After fifteen minutes add 30 cubic centimeters of ammonia solution of den- y 0·95. Let stand several hours (two hours are usually enough.) Filter, wash with ute ammonia, ignite intensely for ten minutes, and weigh.

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[*] Fully described in Bull. No. 15, p. 32.

[†] Communicated to the author in MSS.

[‡] Bull, No. 19, Chem. Div. U. S. Dep't Ag'l, p. 58.

ESTIMATION OF TARTARIC ACID.

The method used in this estimation was that known as the "Goldenberg Geroment," iich is described in full in *Chemiker Zeitung* 12, 1888, 390. This and other methods the estimation of tartaric acid in crude argol and other raw materials were lately bmitted to a critical comparison in the tartrate factory at Nienburg;[*] the Golden- rg Geromont method avoided the principal sources of error and is recommended as e best and most easy of execution.

The procedure as modified for application to a tartate baking-powder is as fol- ws:—

Weigh out 5 grams., wash into a 500-cubic centimeter flask with about 100 cubic ntimeters of water; add 15 cubic centimeters of strong hydrochloric acid; make up to irk and allow the starch to settle. Filter, measure out 50 cubic centimeters of the ar filtrate; add to it 10 cubic centimeters of a solution of carbonate of potash contain- g 300 grams. K_2CO_3 to the liter and boil half an hour; filter into a porcelain dish and aporate filtrate and washings to a bulk of about 10 cubic centimeters. Add gradually th constant stirring 4 cubic centimeters glacial acetic acid, and then 100 cubic centi- ters of 95 per cent. alcohol, stirring the liquid until the precipitate floating in it assumes crystalline appearance. After it has stood long enough for this precipitate to form and tle, best for several hours, decant through a small filter, add alcohol to the precipitate, ing it on the filter, wash out the dish and finally the filter carefully, with alcohol, til it is entirely free from acetic acid. Transfer filter and precipitate to a beaker, d water and boil, washing out the dish also with boiling water if any of the precipi- te adheres to it. The resulting solution is titrated with decinormal alkali solution ing phenol-phthalein as indicator; 1 cubic centimeter decinormal alkali correspond ·0188 grams. of potassium bitartrate, or ·0150 grams. of tartaric acid.

ESTIMATION OF ALKALIES.

The estimation of the soda and potash in the powders was carried out by separating em as chlorides, determining the potash as potassium platinic chloride and calculating e difference as sodium chloride. The detailed procedure was similar to that used by e Association of Official Agricultural Chemists for determining potash in fertilizers, as llows:

Weigh out 5 grams. into a platinum dish and incinerate in a muffle at a low heat. ie charred mass is well rubbed up in a mortar, then boiled 15 minutes with about 200 bic centimeters of water to which has been added a little hydrochloric acid. The hole is transferred to a 500-cubic centimeter flask and after cooling made up to the irk and filtered. Of the filtered liquid 100 cubic centimeters, representing 1 gram. of e sample, are measured out, heated in the water bath, and slight excess of barium chloride added; then without filtering barium hydrate is added in slight excess, the precipitate filtered off and washed. To the filtrate is added a little ammonium hydrate and then ammonium carbonate until all the barium is precipitated. This precipitate is filtered and washed, the filtrate evaporated to dryness and carefully ignited until all volatile matter is driven off, when it is weighed. This gives the weight of the mixed chlorides. The residue is taken up with hot water, from 5 to 10 cubic centimeters of a 10 per cent. solution of platinic chloride added, and the whole evaporated to a syrupy consistence in the water bath; then it is treated with strong alcohol, the precipitate washed with alcohol by decantation, transferred to a Gooch crucible, dried at 100° C., and weighed. The weight of the precipitate multiplied by ·19308 gives the weight of K_2O, and by ·3056 the equivalent amount of KCl. The weight of KCl found is subtracted from the weight of the mixed chloride, the remainder being the NaCl, which multiplied by ·5300 gives the weight of Na_2O in the sample.

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[*] Chemiker Zeitung 13, 1889, 160.

ESTIMATION OF ALUMINIUM.

In the case of a "straight" alum powder, the simple estimation by burning to ash, extracting, and determining the alum by direct precipitation with ammonia would probably be accurate, but in view of the frequent use of flour as a "filler," as well as of the presence of calcium as an impurity, it is best, even with those made up with alum alone, to use a method which will insure a complete separation of the alum. The following procedure, given by Allen for the quantitive estimation of alum in bread, was found to give good results with baking-powders:

Take 5 grams. and incinerate in a muffle, The heat should be moderate so as not to fuse the ash. The process is completed by adding pure sodium carbonate and a little nitre, and heating the mixture to fusion. The product is rinsed out with water into a beaker, acidulated with hydrochloric acid, and evaporated to dryness. The residue is taken up with dilute acid, the liquid made up to 500 cubic centimeters in a graduated flask, filtered, and 50 cubic centimeters taken for precipitation. To the solution dilute ammonia is added until the precipitate barely redissolves on stirring, when a slightly acid solution of ammonium acetate is added, and the liquid brought to a boil. After a few minutes' heating the solution should be set aside for some hours, when its appearance should be observed. (If gelatinous it probably consists solely of iron and alum phosphates, but if granular more or less of the earthy phosphates have been co-precipitated; then it should be separated, redissolved in dilute hydrochloric acid, the solution again neutralized with ammonia, and treated with ammonium acetate.) The precipitate of iron and aluminium phosphates is filtered off, washed, and redissolved in the smallest quantity of hydrochloric acid. The resultant solution is poured into an excess of an aqueous solution of *pure* caustic soda contained in a platinum or nickel vessel. After heating for some time, the liquid is considerably diluted and filtered. The filtrate is acidulated with hydrochloric acid, ammonium acetate and a few drops of sodium phosphate added, and then a slight excess of ammonia. The liquid is kept hot till all smell of ammonia is lost, when it is filtered, and the precipitated aluminium phosphate washed, ignited, and weighed. Its weight multiplied by 3.713 gives the ammonia alum (hydrated), or by 3·873 the potash alum in the 5 grams. of sample taken.

In the phosphate and alum powders the above method gave a fairly good separation of the alum, but the following separation by means of molybdenum was found to be more exact, and at the same time much more convenient of application.

Weigh out 5 grams. into a platinum dish, char, treat with strong nitric acid, and filter into a 500 cubic centimeter flask. After washing the residue slightly, transfer filter and all back to the platinum dish and burn to whiteness. To the ash add mixed carbonates and fuse. Take up with nitric acid, evaporate to dryness, acidify again with nitric acid, and wash all into the 500 cubic centimeter flask. Nearly neutralize the contents of the flask with ammonia, and add molybdate of ammonium sufficient to precipitate all the phosphoric acid present. Allow to stand some time, make up to the mark, shake thoroughly, and filter off 100 cubic centimeters through a dry filter. This is exactly neutralized with ammonia, keeping the solution as cool as possible to avoid the deposition of molybdic acid. Filter and wash the precipitate, redissolve in dilute nitric acid with the addition of a little ammonium nitrate, and precipitate as before. Filter through a paper filter, burn, ignite, aud weigh as Al_2O_3. The alumina and phosphoric acid may be determined in the same sample by the above method, modifying it as follows: When the solution, ash, etc., have all been brought into the graduated flask, make up to the mark without adding molybdate, filter and take 100 cubic centimeters, nearly neutralize with ammonia, add ammonium nitrate and molybdate of ammonium, digest and filter. The filtrate contains the aluminium and may be precipitated with ammonia as above, while the phosphoric acid is all contained in the precipitate, which may be redissolved in ammonia and precipitated with magnesia mixture.

ESTIMATION OF CALCIUM.

This determination was made as follows:—Weigh out 5 grams. of the sample, transfer to a 500 cubic centimeter flask, add 40 or 50 cubic centimeters of water, and then 20 or 30 cubic centimeters of strong hydrochloric acid. Make up to the mark with water, shake thoroughly, and set aside to allow starch to settle. Filter through a dry filter, and take aliquot parts of the filtrate for precipitation; in phosphate powders not more than 50 cubic centimeters should be used. Nearly neutralize with ammonia, acidify slightly with acetic acid, add ammonia acetate, and boil. Filter from the precipitate, if there be any, add ammonium oxalate, and allow to stand several hours. Filter into a Gooch crucible, and dry at 100°. Weigh as oxalate.

ESTIMATION OF SULPHURIC ACID.

The sulphuric acid was estimated without previous ignition of the powder, as follows:—

Weigh out ·5 to 1 gram. of the powder, according to its character, the former quantity being more convenient for alum powders, and transfer to a beaker. Digest with strong hydrochloric acid until all of the powder, including the starch, goes into solution; add barium chloride to slight excess while still hot, and allow it to stand for twelve hours, or over night. Filter into a Gooch crucible, ignite, and weigh.

ESTIMATION OF AMMONIA.

Ammonia is present either as bicarbonate, or as ammonium sulphate in the alum powders. The estimation was made by the Kjeldahl method, as used by the Association of Official Agricultural Chemists.* Where flour instead of starch is used as a filling the gluten would give ammonia, of course, and wherever a tartrate powder was found to give any appreciable amount of ammonia by the method, a weighed portion was taken, water added, the solution filtered from the starch, and subjected to analysis. The results were practically the same as those obtained directly from the powder. Probably flour is not often used. In the case of the alum powders, the difference that would be made by flour filling was disregarded, as the amount of alum present is sufficiently established by the percentage of aluminium oxide and sulphuric acid found; the amount of ammonia found was almost invariably low in proportion to these other constituents of ammonia alum.

ESTIMATION OF MOISTURE.

The percentage of water of association and combination as given in the analyses was obtained by difference. A number of attempts were made to estimate it directly in the following way: A weighed portion of the sample was placed in a U tube, which was kept immersed in boiling water. At one end this tube was connected with a series of sulphuric acid wash-bottles, and at the other with weighed potash bulbs filled with sulphuric acid, and beyond these with an aspirator. In this way a current of dried air was drawn through the sample while it was kept at 100° C., and the water drawn from it in this way was absorbed by the sulphuric acid in the potash bulb, while the carbonic acid was drawn into the aspirator. The increase in weight of the potash bulbs gave the weight of water absorbed. It was found, however, that the sample would cake into a hard mass, through which a channel would form which would permit the passage of the current of dry air, without affecting the greater mass of the powder, and no exact results could be obtained. Some improvement was made by mixing the powder with dry oxide of zinc, so as to prevent the formation of a channel, but still the results were not at all satisfactory, and the attempt to make a direct estimation was finally abandoned. Even if the determination could be made exact, it is doubtful if all the water of combination could be obtained at 100° C., especially in phosphate and alum powders, and probably a temperature high enough to accomplish this would effect a decomposition of the starch.

EXPRESSION OF THE RESULTS OF ANALYSIS.

The results of analysis are given, first, as acid and basic radicals in percentage composition, while in the second part of each table an attempt has been made to combine these into salts showing the constitution of the powder. The difficulties attending this calculation of the probable combination of the acids and bases were so great that I was frequently tempted to give it up entirely and state only percentage composition. I finally concluded to insert the calculation with the proviso that it should be considered at best merely an approximation to the exact composition of the powder. The obstacles in the way of an exact calculation may be stated as follows: In the first place the amount of total carbonic acid found is always less than that required to form bicarbonate of soda with the amount of sodium oxide found. This is undoubtedly due to a partial action of the acid constituent of the powder upon the bicarbonate, by which carbonic acid has been

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* Fully described in Bull. No. 19. Chem. Div. U. S. Dep't Agriculture.

. The percentage of bicarbonate is therefore calculated from the per cent. of car- ic acid found, and the excess of sodium oxide left is stated as "residual" sodium de, without attempting to make further hypotheses as to the results of its combination h the acid constituent. It is possible that part of the bicarbonate may become con- ted into the normal carbonate under the conditions of being mixed and in contact h other chemicals, though this is not likely; then the indefinite composition of many the commercial salts used in the powders renders it an extremely difficult matter to ive at any satisfactory conclusion as to the make up of the powder in which they are d. The acid phosphate of lime, for instance, is a very variable substance, and even nonia alum, which might reasonably be supposed to be constant in its composition, is nd to vary widely from the theoretical. Its content of water varies according to the ater or less amount of drying it has undergone, and aside from this the ratios of the nonia, sulphuric acid, and alumina to one another are at variance with the nula.

This is shown by the following analysis of a sample of commercial ammonia alum ained in a powdered condition:

Analysis of commercial ammonia alum.

Constituents.	Found.	Theoretical Composition.
	Per cent.	*Per cent.*
iminium oxide, $A1_2O_3$	12·62	11·36
phuric Acid, SO_3	34·17	35·28
imonia, NH_3	2·75	3·75
ter of crystallization (by difference)	50·46	49·61
	100 00	100·00

This indicates that the commercial salt is somewhat basic as regards the alumina, there is a deficiency of ammonia, so that if the former is all combined with sulphuric l as normal sulphate, there is still not sufficient of the acid left for combination with ammonia, although the latter is present in too low a proportion to the other consti- nts. This anomaly holds good throughout many of the samples.

I have expressed the sum total of the alumina, sulphuric acid, and ammonia as ıhydrous ammonia alum," combining the sulphuric acid, first with the alumina as far t went, and the rest with the ammonia, and where there was not sufficient for com- ation with all the ammonia adding the latter *as ammonia*.

The presence of acid phosphate of lime still further complicates this calculation, as a question how much of the sulphuric acid should be taken from the alum to com- with part of the lime as sulphate of lime.

In the expression of the results where acid phosphate of lime is present, I have bined the lime with phosphoric acid as normal phosphate as far as it went, and ed the rest as free phosphoric acid, grouping the whole together and calling it "acid sphate of lime." Following is an analysis of a sample of commercial acid phosphate ime, obtained from the trade:

Analysis of commercial acid phosphate of lime.

	Per cent.
Calcium Oxide, CaO	24·93
Phosphoric acid, P_2O_5	52·45
Sulphuric acid, SO_3	.15
Water	22·80
	100·33

In this sample the ratio of lime to phosphoric acid is about 1 : 2, and this relation holds good in many of the powders containing the phosphate, but in some it is quite different. The above sample is almost free from sulphate of lime, while many of the powders show considerable quantities of it, indicating that all the acid phosphates are not so pure in this respect. Chemists will readily understand the impossibility of giving the proportions of the various forms of lime phosphates contained in such a substance. As given in the tables the relative acidity is shown, though of course the phosphoric acid does not occur as free acid.

Both the alum and the lime phosphate contain large percentages of water, hygroscopic moisture in the latter, and crystallisation water in the former, so that the per cents. of the "anhydrous" salts given are always lower than the proportions of the hydrated salts originally used in compounding the powder. Nearly half the weight of the alum is crystallisation water, some part of which is probably driven off in some cases when it is used for baking-powder purposes, but there are no means of ascertaining how much, and of course the moisture in the acid phosphate would vary in different samples, so there is no possible way of approximating the amounts of the hydrated substances, as they were originally used.

Ammonia bicarbonate is another substance of indefinite composition. As given in the tables, it has been calculated from the ammonia found, upon the assumed composition given it in the U.S. Pharmacopœia, viz., $NH_4HCO .NH_4NH_2CO_2$.

In case ammonia carbonate were present in any of the powders containing ammonia alum, I know of no way of estimating the amount or even the fact of its presence in the small quantities used.

The percentage of "available carbonic acid" is placed first in the tables as constituting the most important indication of the efficiency of the sample as an aerating agent.

ON THE DENSITIES AND REFRACTIVE INDICES OF CERTAIN OILS.

By J. H. Long.

(Concluded.)

Olive Oil, 1.

T.	D.	N.	$\frac{N-1.}{D}$
18	·9141	1·4702	·5144
19	·9134	1·4698	·5143
20	·9127	1·4695	·5143
21	·9121	1·4691	·5143
22	·9114	1·4688	·5144
23	·9107	1·4684	·5143
24	·9101	1·4680	·5142
25	·9094	1·4677	·5143
30	·9061	1·4658	·5142
35	·9028	1·4640	·5140

Olive Oil, 11.

T.	D.	N.	$\frac{N-1.}{D}$
18	.9139	1·4719	·5164
19	·9133	1·4715	·5163
20	·9126	1·4711	·5162
21	·9119	1·4707	·5162
22	·9112	1·4703	·5161
23	·9106	1·4698	·5159
24	·9099	1·4694	·5159
25	·9093	1·4690	·5158
30	·9060	1·4669	·5153
35	·9027	1·4648	·5148

Olive Oil, 10.				*Olive Oil,* 5.		
D.	N.	$\frac{N-1.}{D}$	T.	D.	N.	$\frac{N-1}{D}$
·9142	1·4718	·5161	18	·9149	1·4704	·5142
·9135	1·4714	·5160	19	·9142	1·4701	·5142
·9129	1·4709	·5158	20	·9136	1·4697	·5141
·9122	1·4705	·5158	21	·9129	1·4693	·5141
·9115	1·4701	·5157	22	·9122	1·4690	·5141
·9109	1·4696	·5155	23	·9116	1·4685	·5140
·9102	1·4692	·5155	24	·9109	1·4682	·5140
·9095	1·4688	·5154	25	·9103	1·4678	·5139
·9062	1·4666	·5149	30	·9069	1·4659	·5137
·9029	1·4645	·5148	35	·9036	1·4640	·5135

Cotton-seed Oil, 6.				*Cotton-seed Oil,* 8.		
D.	N.	$\frac{N-1.}{D}$	T.	D	N.	$\frac{N-1.}{D}$
·9199	1·4735	·5147	18	·9199	1·4749	·5163
·9192	1·4731	·5147	19	·9193	1·4745	·5162
·9186	1·4727	·5146	20	·9186	1·4741	·5161
·9179	1·4724	·5147	21	·9179	1·4737	·5161
·9172	1·4720	·5147	22	·9172	1·4733	·5160
·9165	1·4716	·5146	23	·9165	1·4729	·5160
·9158	1·4712	·5145	24	·9159	1·4725	·5159
·9151	1·4709	·5147	25	·9151	1·4721	·5159
·9118	1·4690	·5144	30	·9118	1·4700	·5155
·9084	1·4671	·5142	35	·9084	1·4680	·5152

Cotton-seed Oil, 7.				*Cotton-seed Oil,* 9.		
D.	N.	$\frac{N-1}{D}$	T.	D.	N.	$\frac{N-1.}{D}$
·9224	1·4729	·5127	18	·9201	1·4737	·5148
·9217	1·4725	·5127	19	·9194	1·4733	·5148
·9210	1·4722	·5127	20	·9188	1·4730	·5148
·9203	1·4718	·5127	21	·9180	1·4726	·5148
·9196	1·4715	·5127	22	·9174	1·4722	·5147
·9190	1·4711	·5126	23	·9167	1·4718	·5147
·9183	1·4708	·5127	24	·9160	1·4714	·5146
·9176	1·4704	·5127	25	·9154	1·4710	·5145
·9142	1·4687	·5127	30	·9119	1·4690	·5143
·9109	1·4670	·5127	35	·9085	1·4671	·5141

Cotton-seed Oil, 12.				*Sesamé Oil,* 4.		
D.	N.	$\frac{N-1}{D}$	T.	D.	N.	$\frac{N-1.}{D}$
·9203			18	·9208	1·4742	·5150
·9196			19	·9201	1·4738	·5149
·9190			20	·9194	1·4735	·5150
·9183	1·4708	·5127	21	·9188	1·4731	·5149
·9176			22	·9181	1·4727	·5149
·9170	1·4700	·5125	23	·9174	1·4723	·5148
·9163			24	·9167	1·4719	·5148
·9157			25	·9161	1·4715	·5147
·9124			30	·9127	1·4696	·5145
·9090			35	·9093	1·4677	·5144

Cotton-seed Oil, 16.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9203	1·4747	·5158
19	·9196	1·4743	·5158
20	·9189	1·4739	·5157
21	·9182	1·4735	·5157
22	·9176	1·4731	·5156
23	·9168	1·4727	·5156
24	·9162	1·4723	·5155
25	·9155	1·4719	·5155
30	·9120	1·4699	·5153
35	·9086	1·4679	·5150

Sesamé Oil, 14.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9212	1·4756	·5163
19	·9206	1·4752	·5162
20	·9199	1·4748	·5161
21	·9192	1·4744	·5161
22	·9185	1·4740	·5161
23	·9179	1·4736	·5160
24	·9172	1·4732	·5159
25	·9165	1·4728	·5159
30	·9132	1·4708	·5156
35	·9098	1·4688	·5153

Sesamé Oil, 18.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9195	1·4744	·5159
19	·9188	1·4740	·5159
20	·9181	1·4736	·5158
21	·9174	1·4732	·5158
22	·9167	1·4728	·5158
23	·9160	1·4724	·5157
24	·9154	1·4720	·5157
25	·9147	1·4716	·5156
30	·9113	1·4695	·5152
35	·9078	1·4675	·5150

Mustard Oil, 15.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9140	1·4754	·5201
19	·9133	1·4750	·5201
20	·9127	1·4746	·5200
21	·9120	1·4742	·5200
22	·9113	1·4738	·5199
23	·9107	1·4733	·5197
24	·9100	1·4730	·5198
25	·9094	1·4726	·5197
30	·9060	1·4705	·5193
35	·9028	1·4685	·5189

Mustard Oil, 2.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9129	1·4749	·5203
19	·9123	1·4745	·5201
20	·9116	1·4741	·5201
21	·9109	1·4737	·5199
22	·9103	1·4733	·5199
23	·9097	1·4729	·5198
24	·9090	1·4725	·5198
25	·9084	1·4722	·5198
30	·9052	1·4702	·5194
35	·9020	1·4683	·5192

Mustard Oil, 17.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9132	1·4748	·5199
19	·9125	1·4744	·5199
20	·9119	1·4740	·5198
21	·9112	1·4736	·5198
22	·9106	1·4732	·5197
23	·9099	1·4728	·5196
24	·9093	1·4724	·5195
25	·9086	1·4721	·5196
30	·9053	1·4701	·5193
35	·9021	1·4681	·5189

Castor Oil, 3.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9602	1·4799	·4998
19	·9595	1·4795	·4997
20	·2589	1·4791	·4996
21	·9582	1 4788	·4997
22	·9575	1·4784	·4996
23	·9569	1·4780	·4995
24	·9562	1·4777	·4996
25	·9555	1·4773	·4995
30	·9522	1·4755	·4994
35	·9488	1·4738	·4994

Lard Oil, 13.

T.	D.	N.	$\frac{\text{N}-1}{\text{D}}$
18	·9137	1·4694	·5137
19	·9130	1·4690	·5137
20	·9122	1·4686	·5137
21	·9116	1·4682	·5136
22	·9109	1·4678	·5136
23	·9102	1·4674	·5135
24	·9095	1·4670	·5135
25	·9088	1·4666	·5134
30	·9053	1·4647	·5133
35	·9019	1·4627	·5130

Peanut Oil, 19.

T.	D.	N.	$\frac{N-1}{D}$
18	·9187	1·4725	·5143
19	·9180	1·4721	·5143
20	·9173	1·4717	·5142
21	·9164	1·4713	·5143
22	·9157	1·4709	·5143
23	·9151	1·4705	·5142
24	·9144	1·4701	·5141
25	·9138	1·4696	·5139
30	·9103	1·4678	·5139
35	·9069	1·4658	·5136

The numbers in the column headed $\frac{N-1}{D}$ are not quite constant, but do not vary ore than is the case with many organic substances. In most cases the refractive index eems to decrease somewhat more rapidly than does the specific gravity.

The saponification equivalents found for several of these oils, sesamé, olive, and tton-seed, show a mean molecular weight of about 870. From this the *molecular* fractive energy, $M\left(\frac{N-1}{D}\right)$, is about 448, which coincides very well with the atomic fractive energies, C=4·85, H=1·50, and O=3.

If an oil in the above table contained only tri-olein these numbers would lead to 50·4. A mixture of tri-olein and tri-stearin would yield nearly the same result; while i-palmitin, having a lower molecular weight, would make the number slightly smaller. mixture of 8 parts of tri-olein and 2 parts of tripalmitin would give 447·2, and this approximately the composition of the above oils.

In the olive oils the specific gravity at 20° C. varies between ·9126 and ·9136. hese values are about the same as commonly given for the finest cold-pressed oils, and e within the limits of the United States and British Pharmacopœias. Schaedler ves ·9143 as the specific gravity of a pure sample at 20°, but states that in hot-pressed ls rich in palmitin it may be as high as ·922 at the same temperature. The refractive dices at 20° vary from 1·4695 to 1·4711, and the specific refractive energy from ·5143 ·5162 at the same temperature.

In the six samples of cotton-seed oil examined the variations in the specific gravity 20° are between ·9186 and ·9210. These numbers are all greater than those with ive oil. The refractive indices vary from 1·4722 to 1·4741, and the refractive energies tween ·5127 and ·5161 at 20°. These last numbers are nearly the same as with ive oil.

It will be noticed that the values given for the specific gravity are much lower an those usually quoted. Allen places the density of the crude oil between ·928 and 30, with that of the refined between ·922 and ·926. Dietrich (*Dingler's Poly. Jour.*, 85, 127) places the density of the pure oil at 23° between ·917 and ·921. These umbers have evidently been obtained from samples richer in palmitin than were those xamined by me.

In the three samples of sesamé oil the specific gravity at 20° varies between ·9181 and ·9199, the refractive indices between 1·4735 and 1·4748, and the quotient $\frac{N-1}{D}$ between ·5150 and ·5161. These numbers are similar to those for cotton-seed oil.

In mustard oil the density is lower; at 20° the three samples vary between ·9116 and ·9127, their refractive indices between 1·4740 and 1·4746, and the refractive energies between ·5198 and ·5201. These figures are higher than found for other oils, and may have a practical value. In castor oil we have a high gravity and low refractive energy.

The specific gravities found for lard oil and peanut oil do not differ materially from those usually given, and there seems to be nothing unusual about the refraction.

In many cases determinations of refractive index have a practical use in the identification of organic liquids, and also in quantitative valuation.

Alex. Müller has recently proposed the use of the Abbé refractometer in the examination of butter, and Skalweit (*Rept. Anal. Chem.* 1886, pp. 181 and 235) has carried out a number of tests to show the value of the method. It appears from his tests that *butter oleine* (separated after melting by partial cooling and pressure) has a lower refractive index than any oil in the above table, and also lower than that of the various solid products, as butterine and oleomargarine.

Skalweit's observations have undoubtedly a practical value, but his method necessitates the use of the Abbé refractometer. I have found that results almost as sharp can be obtained by using solutions of the fats made with definite mixtures of alcohol and chloroform, and determining the minimum deviation in the usual manner with the Meyerstein or similar instrument. The above experiments show that a determination of the refractive power, coupled with that of the specific gravity, may prove of value in the identification of other fatty oils.

ON THE BEHAVIOUR OF PHENOL-PHTHALEIN WITH AMMONIA.

By J. H. Long.[*]

The peculiar action of ammonia on phenol-phthalein was first pointed out by Beckurts, I believe, in a paper in the *Pharmaceutische Centralhalle.* Soon after the experiments of Luck[†] were published, showing the great value of this substance as an indicator, it came into general use in Germany, and was even incorporated among the tests of a new edition of the German Pharmacopœia then published. Schlickum, in his "Commentary on the German Pharmacopœia," 2nd ed., states in one place that the behaviour of litmus and phenol-phthalein in the titration of acids with ammonia water is the same, while in another place he remarks that the presence of ammonium salts interferes somewhat with the delicacy of the titration when phenol-phthalein is the indicator. Beckurts shows the error into which a number of writers had fallen in this regard, by giving a series of tests carried out with different indicators. In the following year, Flückiger[‡] published experiments on the sensitiveness of phenol-phthalein, leading to the conclusion that it could not be employed with ammoniacal solutions. In the excellent papers by Thomson,[§] a similar conclusion is reached.

In the *Pharmacist* I published the results of a long series of experiments on the action of phenol-phthalein, and among other points, I touched on the want of delicacy with ammonia. In that paper I offered an explanation of this behaviour, suggested in part by a remark of Baeyer[*] on the formation of phenol-di-imido-phthalein, d partially confirmed this explanation by a number of experiments. Since then I ve again taken up the subject, repeating the old experiments and adding new ones, hich I think afford a full confirmation of my former suggestion. It is this later work hich I wish to present here in detail.

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[*] *American Chemical Journal.*

[†] Ztschr. anal. Chem. 1877, 332.

[‡] Ber. d. chem. Gessell. 17, 592. Ref.

[§] *Chem. News*, 47, 125, 135 and 184.

[*] Ber. d. chem. Gessell. 11, 1297.

When a few drops of an alcoholic solution of phenol-phthalein are added to a strong lution of soda, the deep red colour which forms at first, vanishes in a short time— ithin a minute, if the soda is sufficiently strong. The colour can be restored by partial utralisation with acids. When the indicator is added to strong ammonia solution 6 per cent), a red colour is produced which is more permanent, fading completely only ter hours or days.

In this case, that is, after the *complete* disappearance of the colour, addition of acid roduces no change. If the acid is added before the colour is fully discharged it becomes uch brighter.

When phenol-phthalein is added to a very weak soda solution, the red colour pro- uced seems to be indefinitely permanent; but with weak ammonia it vanishes sooner or ter, the interval between the mixing of the solutions and the disappearance of colour pending on several factors, as dilution, temperature, and strength of indicator.

There are, therefore, important differences between the action of soda and that ammonia on phenol-phthalein. If to a definite amount of a standard solution of hydro- loric acid containing a few drops of the indicator, standard ammonia solution be added, mething more than the theoretical amount of the latter must be used to give the ermanent red colour. However, by increasing the amount of indicator added, or by oling the solutions, the colour can be obtained by nearly or quite the equivalent umber of c.c. of ammonia.

A similar result is obtained by adding soda solution to a solution containing, along ith free mineral acid, some salt of ammonium and the indicator. The effect of tem- rature was shown by the following experiments. I prepared half normal solutions of ydrochloric acid and sodium hydroxide and a solution of ammonium chloride containing 0 grms. in one litre. I took now 25 c.c. of the acid, 10 c.c of the ammonium loride, 65 c.c. of water, and a few drops of the indicator. To the mixture, slightly ss than 25 c.c. of the sodium hydroxide was added, the whole was cooled to the desired mperature, and then more sodium hydroxide was run in to colouration. The results tained at various temperatures are given in this table :—

Temperature.	c.c. $\frac{N}{2}$ NaOH.	Temperature.	c.c. $\frac{N}{2}$ NaOH.
6·5	26·2	49·5	29·9
13·5	27·0	62·0	31·0
22·5	27·6	69·1	32·0
30·0	28·1	76·0	32·8
38·0	28·7		

The final reaction was in no case as sharp as in a titration between the alkali and id alone, but the results are close approximations. Something similar is shown in the llowing experiments, which disclose likewise the effects of excess of ammonium loride. Here I employed normal solutions of hydrochloric acid, sodium hydroxide, d ammonium chloride, and for each experiment took 25 c.c. of acid, a definite quan- ty of ammonium chloride, and added enough water to make 100 c.c. In each case c.c. of a one-tenth per cent. solution of phenol-phthalein was added, and then caustic da from a burette to colouration.

The solution in the beaker was kept at a temperature of 0° C., and that in the urette as near this as possible. The experiments were repeated for each mixture three mes, and gave closely agreeing results, the end reactions being quite satisfactory and arp. In the following table the mean of the results from the three tests is given :—

	HCl.	NH_4Cl.	H_2O.	NaOH req'd.
No.	c.c.	c.c.	c.c.	c.c.
1	25	5	70	25
2	25	10	65	25·08
3	25	15	60	25·20
4	25	25	50	25·28
5	25	30	45	25·37
6	25	50	25	25·68

These experiments show clearly that with the temperature sufficiently low, the disturbing action of small amounts of ammonium salts is very slight. They show also at what rate the disturbance increases with increasing amounts of ammonium chloride.

At a temperature of 20° C. I obtained the following results, working with the same solutions:—

	HCl.	NH_4Cl.	H_2O.	NaOH req'd.
No.	c.c.	c.c.	c.c.	c.c.
1	25	5	70	25·22
2	25	10	65	25·38
3	25	15	60	25·61
4	25	20	55	25·82
5	25	25	50	25·98

We find here differences in the amount of alkali required, varying between ·2 c.c. for the weakest solution to 1 c.c. for that containing 25 c.c. of ammonium chloride, which amounts to an error of 4 per cent. In all these cases the error is so large that it could not be neglected in any kind of practical work. However, by employing a larger amount of indicator, the error can be almost entirely corrected, as shown by the following tests:—

I used here 25 c.c. of the acid and 5 c.c. of the ammonium chloride solution at a temperature of 20° C.

1. With 2 c.c. of indicator required 25·12 c.c. NaOH.
2. With 4 c.c. of indicator required 25·02 c.c. NaOH.
3. With 6 c.c. of indicator required 25·00 c.c. NaOH.

By using larger amounts of the indicator, the final reaction is obtained with satisfactory sharpness. *(To be continued.)*

REVIEWS.

WATT'S MANUAL OF CHEMISTRY THEORETICAL AND PRACTICAL (BASED ON FOWNES' MANUAL). VOL. I. PHYSICAL AND INORGANIC CHEMISTRY, SECOND EDITION, BY WILLIAM A. TILDEN, D.SC., F.R.S. LONDON: J. AND A. CHURCHILL.

IN this edition we welcome the appearance of what is really the fourteenth edition of our old friend "Fownes," from the pages of which most of our living chemists have received their primary instruction. The new Editor has done his part in a very careful manner and has succeeded in bringing the work up to date without any undue increase in size. Another feature for which he is responsible is the doing away with the separate introductory physical part and the scattering of the same through the text. In this way, when it is necessary to refer to any physical point in connection with the chemical subject in hand, the matter in question is then and there fully explained. Of course no chemical book is quite free from printers' errors, and when they occur in this edition they are sufficiently glaring, as for example, when we are told upon page 9 that "$3HCl$ or $(HCl)_3$ denotes 2 molecules of hydrogen chloride." Taking, however, the book as a whole, there is really no other work extant that occupies so useful a middle place between the cram book and the great works of reference as does this work, of which it might be said that authors come and go but "Fownes" goes on for ever.

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MARCH, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of the Society was held at Burlington House, on the 12th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members: Robert Hellon, Ph.D., Public Analyst for Cumberland, and J. B. Harrison, of British Guiana.

The following papers were read:—

"On the Composition of Milk and Milk Products."—By Dr. P. VIETH.

"On the Detection of Methylated Sweet Spirits of Nitre."—By Dr. MUTER.

"On the Detection of Cotton Seed Oil in Lard."—By Dr. MUTER AND L. DE KONINGH.

"On the Solubility of Phosphate of Alumina in Acetic Acid, with special reference o the extraction of Alumina from Flour, Bread, etc."—By W. C. YOUNG.

A paper by Dr. Muter "On the Analysis of Disinfecting Powders Containing Commercial Carbolic and Sulphurous Acid," was deferred till next meeting.

The next meeting will be held at Burlington House, on Wednesday, the 12th inst.

THE ANALYSIS OF MEDICINES DISPENSED FROM PHYSICIANS' PRESCRIPTIONS: A RAPID METHOD OF ESTIMATING QUININE IN MEDICINE.

By Dr. Seaton and Mr. H. Droop Richmond.

(*Read at the Meeting, January,* 1890.)

The analysis of medicines dispensed from physicians' prescriptions is a part of the work of a public analyst which has not, as yet, received the amount of attention it deserves. This seeming neglect is due to several causes, the chief of which seems to be the difficulty in obtaining the effective combination of the several persons whose combined action is required. To procure a sample of medicine which may be submitted to the public analyst under the Food and Drugs Adulteration Act, it is necessary, first of all, that the inspector appointed under the Act should in this particular, be under the immediate guidance of the Medical Officer of Health; then a physician's prescription (clearly written) must be obtained, which is suitable for analysis; and thirdly, the sample when obtained under the provisions of the Act, must be submitted to the Public Analyst, who very frequently has no immediate communication either with the authority whom he serves or its officers. In writing a prescription for the purpose of obtaining a sample of medicine for analysis, regard must be had not only to the suitability of the drug—simple or combined, from the analytical point of view—but to its exhibition in forms or doses usually prescribed by English practitioners in medicines. For example, if an inspector were to present a prescription for a quinine mixture containing more than 2 to 5 grains in a dose, the attention of the dispenser would immediately be directed to the unusual character of the prescription, and consequently more than usual care would be taken in the dispensing of the medicine. In taking the sample under the Act the inspector has to divide the medicine into three parts, and consequently the amount of the drug present in the third part submitted to the Public Analyst is frequently very small.

A not uncommon prescription for a quinine mixture is as follows:—

Quiniæ sulphatis, gr. xii.
Acid : sulph : dil. ♏ xx.
Aquæ destillatæ, ℥ viii.

Here the amount of the drug present in a third part of the medicine is only 4 grains of the sulphate (the alkaloid being, of course, still less), so that the operation of analysis must be very delicate if errors in dispensing of such amounts as 10 per cent. or 20 per cent. are to be estimated. Our late President, Mr. Allen, was the first to take up this subject. His action must have produced a good effect, not only in the neighbourhood of Sheffield, but throughout a far wider sphere. In discussing some of the results of Mr. Allen's work, Dr. Dupré remarked that his own experience showed that a considerable amount of adulteration was practised in relation to drugs and medicines—that is to say, if the leaving out of an appreciable proportion of the active agent is to be considered adulteration. Most certainly it is, and we would further say that the *adding* of an appreciable proportion of the active agent would, we believe, be considered an adulteration. A short time since we had occasion to analyse three samples of medicines containing bromide of potassium as the active agent. In one sample we found that the amount present exceeded the amount prescribed by 25 per cent., and we have no doubt t if this case could have been the subject of magisterial proceedings a conviction uld have been obtained.

But to return to Mr. Allen's investigations. We find on the 19th page of THE ALYST, Vol. 1, a paper on the determination of quinine by the ammonia and ether cess, which is generally pursued, but which is a lengthy and difficult process even en carried out by the modification suggested by Mr. Wynter Blyth. It also necessi- es the use of an expensive reagent, viz., ether. These objections to the general ption of the ammonia and ether process have led us to the suggestion of the following thod, which we have found to be perfectly reliable within certain limits, and which the advantage of being rapid and inexpensive. We do not recommend it in substi- ion of the ammonia and ether process, as the final method of determining the portion of quinine in medicine, but as a preliminary process which will enable the lyst to decide whether an error in dispensing has been committed, sufficient in extent bring the case within the scope of the Adulteration Act. We have found that inine bisulphate is neutral to methyl orange, while the base itself has no action on enol phthalein; and we have, bearing these facts in mind, adapted to the determina- n of quinine the method of alkalimetry. We prefer to operate as follows:—

To 25 c.c. of medicine add 2 drops of methyl orange (·25 grm. in one litre of water), d 2 drops of phenol phthalein (·5 grm. in one litre of 50 per cent. alcohol), titrate with $\frac{N}{10}$ ryta solution until the free acid is all neutralised, which is shown by the red colour st changing to a brown. Note the number of c c. used. Continue titration until the k colour of the phenol phthalein appears. Care must be taken not to overstep is last point, as the colour does not develope quite immediately. The difference tween the two titrations, multiplied by the factor ·0218, gives the weight of the inine sulphate (*i.e.*, the 7-molecule of water hydrate) in grms. in the 25 c.c. The mber of c.c. in the medicine prescribed is known, and the calculation becomes a nple proportion sum.

The method is, of course, not applicable in the presence of salts, of which the base precipitated by Baryta.

The following is the result of the estimation of quinine in the same medicines by e volumetric and gravimetric processes respectively. Four samples of medicine were tained in the usual way, and it will be seen that the results of the two methods sely correspond:—

	Volumetric.	Gravimetric.
1.	21·2	20·8
2.	23·4	23·2
3.	16 5	16·1
4.	20·3	20·0

We have further tested the process in the following way: Solutions of quinine, curately prepared, were obtained by one of us from Messrs. Bell and Co. Solution A ntained 12 grains by weight of Howard's quinine sulphate; solution B, 20 grains; lution C, 18 grains; and solution D, 15 grains. Four samples of each solution were tained, and they were submitted to the other for estimation by our process.

The process gave the following results:—

A. (12 gr.) found respectively	12·1	12·2	11·8	12·0
B. (20 gr.) ,,	19·7	19·9	19·9	19·7
C. (18 gr.) ,,	17·5	17·6	17·5	17·8
D. (15 gr.) ,,	15·4	14·7	14·9	15·2

ON THE COMPOSITION OF MILK AND MILK PRODUCTS.

BY DR. P. VIETH.

(*Read at Meeting, February,* 1890.)

THE kind reception which you have accorded to former communications of a similar kind (see THE ANALYST, VII. p. 53; VIII. p. 33; IX. p. 56; X. p. 67; XI. p. 66; XII. p. 39; XIII. p. 46; and XIV. p. 69) encourages me to lay before you to-night what perhaps might be best described as "a report on the work done in the laboratory, which is under my charge, during the year 1889."

The fact that the work is carried on from year to year under similar conditions, and with the same objects in view, makes it impossible to avoid in these annual reports a certain uniformity of outside appearance and repetitions with regard to their contents. When first brought before the Society, these reports were considered not without value, and I contend that their continuation cannot deduct from their value, although it may make them less attractive.

The total number of samples submitted to analytical examination during the past year is 22,728, viz.:—

20,674	samples of	milk,
1,123	„	cream,
609	„	skim milk,
29	„	buttermilk,
231	„	butter and butter-fat,
12	„	milk preparations for infants and invalids,
42	„	water,
8	„	sundry articles.

Of the milk samples, 12,617 were taken from the railway churns on their arrival in the dairy, 1,757 from the milk before being sent out, 2,623 by the inspectors employed by the business, from the man while distributing the milk, and 324 from the small quantities of milk brought back from the rounds. The following table contains the monthly averages of the results referring to these samples:—

AVERAGE COMPOSITION OF MILK.

1889.	Samples taken.						
	On arrival.				Before sent out.	During delivery.	From returns.
	Spec. Grvty.	Tot. Sol.	Fat.	Sol. n. fat.	Tot. Sol.	Tot. Sol.	Tot. Sol.
January ..	1·0320	12·79	3·73	9·06	—	12·8	—
February ..	1·0321	12·76	3·70	9·06	—	12·7	—
March ..	1·0319	12·73	3·70	9·03	12·6	12·6	—
April ..	1·0319	12·68	3·66	9·02	12·6	12·6	—
May ..	1·0322	12·67	3·60	9·07	12·6	12·6	—
June ..	1·0322	12·55	3·49	9·06	12·5	12·6	—
July ..	1·0319	12·65	3·65	9·00	12·5	12·6	—
August ..	1·0320	12·77	3·72	9·05	12·7	12·7	12·7
September ..	1·0321	12·91	3·80	9·11	12·8	12·9	12·8
October ..	1·0323	13·21	4·03	9·18	13·1	13·2	13·3
November ..	1·0321	13·19	4·04	9·15	13·1	13·1	13·3
December ..	1·0322	13·02	3·88	9·14	12·9	13·0	13·0
Yearly average	1·0321	12·83	3·75	9·08	—	12·8	—

It will be seen from this table that the richest milk was, as usual, received during the latter part of the year, while the lowest percentages of total solids and fat were observed in the month of June. I attribute the somewhat exceptional quality of the milk received in June—the lowest monthly average observed since the last five years—to the extraordinary atmospheric conditions, to wit, the long spell of warm and dry weather which reigned in May and June. The agreement between the several sets of samples is satisfactory, considering that not much time is allowed for mixing the milk and drawing the samples.

There were further analysed, 1,814 samples of milk, yielded by individual cows. An investigation regarding the composition of milk of individual cows was commenced in April, 1887, and up to the end of last year about 5,000 samples of such milk have been submitted to analysis. It is my intention to bring the results of this investigation under your notice as soon as I shall find the time to work the vast material into a suitable shape.

Cream samples were taken before the cream was sent out, and also while it was delivered to the customers; 412 samples of the former and 303 samples of the latter kind having been analysed with the following results:—

AVERAGE PERCENTAGE OF FAT IN CREAM.

1889.	Samples taken.	
	Before sent out.	During delivery.
January	46·5	47·0
February	46·8	47·4
March	47·2	47·6
April	49·4	48·9
May	49·5	49·9
June	48·2	48·0
July	45·6	45·5
August	45·6	45·7
September	45·6	45·8
October	47·9	47·5
November	48·3	48·9
December	46·8	46·8
Yearly average	47·3	47·4

I have mentioned on former occasions that an absolute agreement between the two sets of samples of cream cannot be expected for two reasons; firstly, because samples taken before distribution refer to morning and afternoon delivery, while samples taken during distribution refer to afternoon delivery only, the cream being left in sealed cans in the morning; and secondly, because there is some difficulty of getting fair average samples from large bulks of cream. Keeping this in mind, I believe the agreement between the two series of analysis is all that can be expected.

The composition of fifty-one samples of clotted cream was as follows:—

Total solids	54·99	to	69·05	average	63·31	per cent.
Fat	47·72	„	62·83	„	56·69	„ „
Solids not fat.. ..	5·37	„	8·47	„	6·62	„ „
Ash	·44	„	·65	„	·53	„ „

I wish, in this place, to draw once more attention to a fact which I have mentioned on several previous occasions, viz., to the close relationship existing in normal milks and creams between the total amount of non-fatty solids and the amount of mineral matter, or ash, contained therein. I have invariably found the ash to amount to, as near as possible, 8 per cent. of the non-fatty solids, and look at this fact not only as a very useful check on the analysis, but also as a proof of the normal quality of a milk.

The 104 samples of butter analysed were of different origin; they may be classed as English, French, Danish and Swedish, and German. Their composition was a follows:—

BUTTER CHURNED IN LONDON, FRESH AND SALT.

Fat	85·62	to	90·49	average	87·13	per cent.
Water	8·03	„	13·48	„	11·55	„ „
Solids not fat ..	·63	„	2·83	„	1·32	„ „
Chlorides, as NaCl ..	·38	„	2·44	„	·96	„ „
Volatile acids, Wollny	25·8	„	29·2	„	26·9	c.c. $\frac{n}{10}$ alk.

FRENCH BUTTER, FRESH.

Fat	83·74	to	85·62	average	84 78	per cent.
Water	13·22	„	14·44	„	13·80	„ „
Solids not fat ..	1·00	„	2 00	„	1·42	„ „
Chlorides, as NaCl ..	.05	„	·17	„	·08	„ „
Volatile acids, Wollny	26·7	„	30 5	„	28·9	c.c. $\frac{n}{10}$ alk.

DANISH AND SWEDISH BUTTER, SALT.

Fat	78·91	to	85·64	average	82·84	per cent.
Water	11·86	„	16·96	„	13·75	„ „
Solids not fat ..	2·21	„	4·70	„	3 41	„ „
Chlorides, as NaCl ..	1·06	„	3·00	„	2·07	„ „
Volatile acids, Wollny	27·2	„	29.8	„	28·3	c.c. $\frac{n}{10}$ alk.

KIEL BUTTER, SALT.

Fat	82·00	to	89·45	average	86·04	per cent.
Water	8·39	„	15·23	„	11·56	„ „
Solids not fat ..	1·70	„	2·89	„	2·40	„ „
Chlorides, as NaCl ..	·84	„	1·81	„	1·29	„ „
Volatile acids, Wollny	21·1	„	29·8	„	26 5	c.c. $\frac{n}{10}$ alk.

The volatile acids were also determined in 127 samples of butter-fat prepared fro small quantities of either milk or cream in the laboratory. The majority of the results these determinations were embodied in a paper which I had the honour of submitting the Society at the meeting in June.

Some pure butter-fat which had been exposed to the action of air and light f one year and nine months, had become quite white. It had been analysed when fresh, and was re-analysed in its bleached condition. The following are the results:—

Fresh.	Bleached.
Insoluble acids, Hehner 87·72 per cent.	Insoluble acids, Hehner 83·82 per cent.
Volatile acids, Reichert 15 0 c.c. $\frac{n}{10}$ alk.	Volatile acids, Wollny 31·7 c.c. $\frac{n}{10}$ alk.

It appears, then, that the insoluble fatty acids decreased at the ratio of 100 to 95 5, while the volatile fatty acids (Reichert's figure doubled) increased at the ratio of 94·6 to 100. The soap and also the insoluble acids were, in the case of the bleached sample, of dark, red-brown colour.

I believe I am right in saying that I have almost every year met with at least one new milk preservative, or rather, with a milk preservative under a new name, for they all have that in common, that they contain as active principle boracic acid in some form or other. "Dr. Beesly's Antiseptic Milk Preservative," which came under my notice last year, proved to be no exception to the rule.

DISCUSSION.

DR. MUTER said it seemed to him that they all ought to be grateful to Dr. VIETH for giving them those figures, because it was the sort of work which enables them to fix accurate limits for things. Curious points often came out, such as that about the relation between solids not fat and ash, a striking matter which could only be discovered by a very large number of analyses being made by one person.

MR. ALLEN asked if Dr. Vieth could guarantee the genuine nature of the exceptional butters to which he referred in his paper.

DR. VIETH, in reply, said he could not guarantee the purity of the Kiel butters not having seen them made himself, but he had not the slightest doubt they were perfectly genuine. The butters came over in casks.

MR. HEHNER asked Dr. Vieth whether it was not a fact that the butter prices had risen at the same time that the volatile acids in Dr. Vieth's samples had declined. He thought that unless the origin of exceptional samples was thoroughly authenticated they should not too readily give evidence to alleged exceptions. He had noticed that soon after the reading of Mr. Allen's paper on the exceptional Danish butters, a great proportion of all butter samples analysed by him had disclosed volatile fatty acids. The foreign shippers of butter knew well how to take advantage of these exceptional samples. Mr. Hehner related several cases from his own experience, which showed that these shippers were now mixing from 10 to 15 per cent. of margarine with butter, in the confident belief that they could not be detected.

MR. ESTCOURT, whilst thanking Dr. Vieth for his valuable paper, said he agreed with Mr. Hehner in his remarks as to the Kiel samples. Mr. Allen had said that these results were not discussable. They certainly did not compare with the Danish and Swedish, which averaged 28 per cent., but he was going to mention a case in which Mr. Allen was himself concerned at a time when mixtures were not supposed to exist. Mr. Allen had the official sample, and he the sample of the vendor.

He agreed with Mr. Allen in pronouncing it to be a mixture.

His clients then came to him and disclosed the name of the Irish firm who sold it them, and it was found that the Irish firm had really been manufacturing this margarine by adding foreign fat to last year's well-washed butter.

He saw the certificate of Dr. Tichborne, of Dublin, stated to be of the same butter, which was also stated to have passed Somerset House. This was given to each purchaser, but the certificates of Mr. Allen and himself put an end to this manufacture, fortunately before Mr. Allen adopted so low a standard as of recent time.

It was quite certain that if they accepted Dr. Vieth's figures, 25 per cent. foreign fat might pass, and they would descend to a lower depth than that.

MR. CASSAL agreed with Mr. Hehner, in thinking that credence was too readily given to these exceptional cases. The position of a public analyst, called upon to make official examinations of butter coming from entirely unknown sources, was, however, very often a difficult one, and it was obvious that the greatest caution should be observed in certifying to small percentages of adulteration. It should be remembered that the average magistrate played a considerable part in prosecutions under the Acts; but more effectual steps might, nevertheless, be taken to check systematic "small" adulteration.

Dr. Vieth, in reply, said the public analyst ought to look at his office from two points of view. While trying to detect adulteration and thus helping to bring offenders to law, he should also think it his duty to protect innocent people. There was no doubt that abnormal butters did exist; these were abnormal samples. Low results had been found in genuine Swedish and Danish butters; they had also been observed in Italy. From a paper of Mr. Allen's, they would remember that Professor Stein in Copenhagen was able to point with his finger to dairies where abnormal butters might be, and indeed were found. He did not think that Mr. Hehner would doubt that genuine butters with abnormally low volatile acids did exist.

A SIMPLE METHOD OF DETECTING THE PRESENCE OF METHYLATED SWEET SPIRIT OF NITRE.

By John Muter, Ph.D., F.R.S.E.

Read at Meeting, February, 1890.

I have never yet met with a really simple test for this fraudulent article mentioned in any print. There is, at the present time a great amount of it about, and it behoves us all to be on the alert to stop it. From what I have seen, the regularly licensed chemists and druggists do not patronise the makers of the article to any great extent, but it chiefly finds its way to cutting stores and oilmen, who undersell the pharmacist in all his articles not included in the poisons schedule. It can, of course, be made of correct specific gravity and to yield the right amount of nitric oxide by the B.P. test, and yet sold at a splendid profit. There ought, certainly, to be introduced into the next B.P. some simple method whereby the stuff can be readily found out in a few moments; and in the hope that this will be done, I beg to submit the following perfectly simple, yet, so far as I know, entirely reliable test. At least, among no end of samples, I have never yet failed with it. The test simply consists in putting some of the sample into a glass, with a lump of solid caustic potash (about the size of a small bean), and stirring till nearly dissolved. Real B.P. spirit, made from rectified spirit of B. P. quality (which it ought to be, or it is not a legal tender as B.P. spirit) will lose all its odour of nitrous ether, and, after standing for half-an-hour, will not have become darker than the very palest tint of straw-colour (not visible by gas-light), and it will then only have the odour of plain rectified spirit. The methylated article, on the other hand, will become of a dark colour, varying from deep yellow to orange red, and will give off the well-known odour of methylated spirit in all its nakedness. Should any farther confirmation be required, then we may have recourse to the old "Miller" process, or we may distill off some of the potashed spirit, and put 10 grams. of it in the dark for 48 hours, with a good excess of Hübl's solution. The genuine B.P. spirit has never, in my hands, absorbed any iodine, while the methylated article takes up from ·4 to ·7 per cent. of iodine, according to the quality of the spirit employed. There is, however, no need to trouble about this, because the simple combined observation of the odour and colour after potash are perfectly convincing.

NOTE ON THE ANALYSIS OF LARD, COTTON OIL, TALLOW, Etc.

By John Muter, Ph.D., F.R.S.E., and L. De Koningh, F.I.C

Read at Meeting, February, 1890.

In the month of February, 1889,* we proposed an improved method for the volumetric estimation of oleic acid in fats and oils, and of subsequently applying Hubl's iodine absorption test to the acid thus liberated, without its having been exposed to heat or to any influence likely to be prejudicial to the accuracy of the iodine number thereby obtained. We feared that the calls of our ordinary business would make it a very tedious matter for us to examine all the common fats and oils, and to suggest standards based upon the process, and we therefore invited our colleagues to give us their co-operation with this object. In the meantime we have been applying it to the current matters that have turned up, and especially to the analysis of lard, and we now give the standards we have adopted in this matter.

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* See Analyst for April, 1889.

1. *Tallow.*—The oleic acid isolated from tallow seems to us to be practically pure oleic acid. In many experiments we have made, we generally get from ·1 to ·2 per cent. from theory. Tallow is therefore, in our opinion, the best source from which to prepare perfectly pure oleic acid, giving an iodine number of 90.

2. *Lard.*—Although it would be unwarrantable to jump to the conclusion that this fat contains acids of the linoleic series, still we have not as yet met with any sample which gives an acid having as low an iodine absorbing power as the oleic acid from tallow. After careful study of many samples we propose to put the iodine number of the oleic acid from lard at 93, but not to condemn under 94.

3. *Cotton Oil.*—This oil being often subjected to a good deal of chemical treatment before it is sold, it would not be a matter of surprise if the iodine number of its oleic acid should be subject to a good deal of variation. However, in all the samples we have as yet tested, we do not find any very great difference, and we propose to put the iodine figure of the cotton oleic acid at 135.

We are not aware that any of our English colleagues have taken up the process, but it has attracted some notice abroad, and just as we are writing, we have received a copy of a paper communicated to the Hungarian Academy of Sciences by Dr. Alex. von Asboth, in which he gives a very favourable criticism of our process, and proposes to fix the iodine numbers of the fluid acids of lard and cotton oil respectively at 94 and 136. Dr. Asboth thus gets the same calculation difference between these two fats as we do, although he is one degree higher than us in both cases, which may after all be only a matter of the "personal equation" in working.

As regards the application of the process it may be interesting to extract two of our experiments on known mixtures, so as to show both the mode of calculating and the degree of accuracy that may fairly be expected in practice.

Experiment A.—This was made on a mixture of 43 per cent. of lard and 57 of cotton oil. The sample yielded 64 per cent. of fluid acids, and these acids gave an iodine absorption figure of 118 (lard = 93, and cotton oil = 135). Therefore (135-93): (118-93) :: 64 : x.x. = 38 cotton oleic acid. As we may take common cotton oil to contain about 70 per cent. of liquid acids, we get 70 : 100 :: 38 : x. x = 54 per cent. of adulteration in the sample.

Experiment B.—Made on a mixture containing 75 per cent. of lard and 25 of cotton oil. Found 58 per cent. of liquid acids which gave an iodine number of 103. Therefore :—(135-93) : (103-93) :: 58 : x. x = 14 per cent. of cotton oil acids. Then as 70 : 100 :: 14 : x. x = 20 per cent. of adulteration.

It will be seen that, like all such processes based upon average standards, the accuracy diminishes as the adulteration gets smaller, and if the latter is below 10 per cent. then the process is only really of qualitative value. However, we agree with von Asboth in saying that any lard the acids of which show a greater iodine absorption than 94, must be looked upon with the gravest suspicion. One thing is certain, namely, that the true imitation lard which, if properly made, can defeat the ordinary iodine test, will not escape detection if treated by our method. The only fat which, if introduced in a judicious quantity, might upset our process is cocoanut oil. This oil contains very little olein, but still yields a fair proportion of acids (chiefly caproic) which also form lead salts that are at once insoluble in water and soluble in ether. Before our process can be applied, it is therefore necessary to remove these acids, and although our investigations on this subject are not yet complete, we believe that the best way to accomplish this will be to heat the apparent oleic acid (obtained by evaporation of the ether) in a current of carbon dioxide at a temperature of 110° to 115° C. until all the interfering acids are removed. After this is done, the remaining acids are to be weighed and "Hubled." The presence of cocoanut fat is so readily detected qualitatively, that no analyst would have any difficulty in condemning a sample containing it, so it is not probable that its use will be resorted to.

We have placed upon the table one of "Muter's olein tubes" as now manufactured in Germany, and which are readily procurable through Messrs. Baird and Tatlock, of Glasgow, or indeed of any similar house.

DISCUSSION.

MR. ALLEN said they were much indebted to DR. MUTER for this continuation of his former very important paper. The process was really one likely to throw light upon the unknown characters of these fatty acids, which rendered it extremely valuable. The constitution of these acids had been up till now ignored by chemists who had too long classed them as being all stearic, palmitic, and oleic. What they had been in the habit of calling oleic acid was by no means of such a constant character as they had been assuming. It was by highly refined researches and methods depending on the constitution of the fatty acids that they could hope to differentiate them and detect the oils of which they were constituents. It was to methods like that of Dr. Muter's and those of Hazura and other recent investigators that they might hope to detect vegetable oils in butter and lard.

(Conclusion of the Society's Proceedings.)

NOTE ON THE PURIFICATION OF ALCOHOL FOR LABORATORY USES

BY E. WALLER, PH. D.

COMPARATIVELY recently several papers have appeared on the subject of the reactions of potassium permanganate with alcohols, and the impurities which they may contain.*

In preparing alcoholic solutions of caustic alkalies, and also of silver nitrate (for fat tests, etc.), I have been annoyed, as have other chemists, by the unsatisfactory character of the solutions obtained in consequence of the presence of impurities in the alcohol bought for laboratory use. I find also that most alcohol of 93 per cent. when kept in tin cans slowly reacts on the tin, giving after a while a cloud of SnO_2 which is too fine to filter out, and renders purification by distillation necessary. In the light of the results obtained by the writers above alluded to, I have adopted the following method for the purification of my alcohol, on which I ask the criticism of the members of the society:—

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* Habermann. Fres. Ztschr. anal. Chem. 27, 663. Roese, ib. 28, 355.
Cazeneuve. Bull. Soc. Chim. (Paris) [3] 1, 700. See also Dingl. Polyt. Jour., 273, 374.

A convenient amount of the alcohol to be purified is shaken with pulverised potassium permanganate until it assumes a decided colour. It is then allowed to stand for some hours, until the permanganate has been decomposed and brown manganese oxide is deposited. A pinch of pulverised calcium carbonate is then added, and the alcohol distilled at the rate of about 50 c. cm. in twenty minutes from a flask provided with a Wurz tube or one of the Lebel-Heninger pattern. The distillate is tested frequently until about 10 c. cm. thereof, when boiled with 1 c. cm. of strong (syrup) solution of caustic soda or potash, gives no perceptible yellow colouration on standing for twenty minutes or half an hour. What distils over after that time is preserved for use.

The first distillates may be added to the small amount remaining in the distilling flask (which should not be driven down to complete dryness), and a fresh portion of purified alcohol recovered.

The rationale of the proceeding appears to be that the permanganate oxidises and destroys chiefly the fusel-oil, furfurol and other compounds of that nature, the acids resulting from the reaction are neutralised by the calcium carbonate added before distillation, and by distilling slowly the aldehyde at least is concentrated in the first portions of the distillate. Distillation of alcohol containing caustic potash or soda seemed to cause a constant formation of aldehyde. The alcohol thus purified is perfectly neutral, and gives most satisfactory results when used as a solvent for caustic alkalies or silver nitrate, the solutions remaining as colourless as distilled water, even after boiling and standing indefinitely, if properly protected from dust and other external influences.—*Jour. Am. Chem. Soc.*

TEST FOR COTTON-SEED OIL IN LARD.

By Frank P. Perkins, F.I.C.

The following may be useful in *sorting* samples of lard which have to be examined for cotton-seed oil.

I find that if a little powdered potassium dichromate—about ·02 to ·03 gramme—be mixed with a few drops of concentrated sulphuric acid in a porcelain dish, and a small portion—about ·5 gramme—of the suspected sample be then introduced, on stirring well a second time, adding water, and stirring again, there will in the presence of cotton-seed oil be developed a green colour, due to the change of chromic acid to chromic oxide, but if the vegetable oil be absent the yellow colour of the dichromate will still prevail. The colour should not be judged until water has been added, and the mixture stirred for some seconds.

This test I have tried with samples of bladder lard, with American lard known to be adulterated, with purely rendered home lard, the same containing a certain proportion of cotton-seed oil, and with cotton-seed oil itself. It has not failed to detect the cotton-seed oil either alone or when mixed with lard. How the test is affected by other vegetable oils I have not yet determined, I therefore submit it with diffidence, and should be glad to know how it answers in the hands of other experimenters.

ON THE BEHAVIOUR OF PHENOL-PHTHALEIN WITH AMMONIA.

By J. H. Long.

(*Concluded.*)

In another series of experiments, ammonia was run into the solution containing hydrochloric acid and different amounts of the phenol-phthalein. Where but a few drops of this were taken, the end reaction could not be observed accurately; with larger amounts the reaction was sharper, but only came after addition of an excess of alkali.

1. With 25 c.c. of acid and 5 c.c. of indicator I used 25·57 c.c. of NH_4OH.
2. With 25 c.c. of acid and 10 c.c. of indicator I used 25·28 c.c. of NH_4OH.

In presence of alcohol the reaction was always less distinct than with water, and more alkali was required to bring it.

The above results seem to indicate that ammonia combines with phenol-phthalein, forming, to some extent, a colourless compound, besides forming a coloured one as does sodium hydrate; and also that this colourless body is produced more quickly as the temperature is higher or the ammonia stronger.

I expressed the opinion in my former paper, referred to above, that the phenol-di-imido-phthalein discovered by Baeyer is the compound formed here, and that this explains the peculiar reaction.

The experiments given below seem to confirm fully this view. Baeyer* states that phenol-phthalein and aqueous ammonia heated to 160°–170° in sealed tubes for some hours react on each other in this way:—

$$C_{20}H_{14}O_4 + 2NH_3 = C_{20}H_{16}N_2O_2 + 2H_2O.$$

The phenol-di-imido-phthalein is a colourless body soluble in alkalies without colour, but which dissolves at a high temperature in acids with formation of an ammonium salt and phenol-phthalein:—

$$C_{20}H_{16}N_2O_2 + 2H_2O + 2HCl = C_{20}H_{14}O_4 + 2NH_4Cl.$$

I prepared the product by the following methods:—

1. 3 grms. of phenol-phthalein and 30 c.c. of strong ammonia were heated during three hours in a sealed tube to 160° C. The tube was then opened and the contents neutralised with hydrochloric acid. This precipitated a gummy mass, which was washed thoroughly with water, and finally dissolved in 90 per cent. alcohol. The solution, after filtration through bone charcoal, was evaporated to dryness. The residue left was dissolved in a mixture of equal parts of benzene and alcohol. The solution was allowed to evaporate spontaneously, leaving a residue, which was heated to 120° C. to completely remove all traces of the solvent. The product then remained as a colourless mass of fine needle-shaped crystals.

2. 10 grms. of phenol-phthalein were treated with 200 c.c. of strong ammonia solution in a flask kept hot on a water-bath during fifteen hours; at the end of that time the solution was neutralised with hydrochloric acid, and an insoluble gummy mass formed was treated as under 1.

3. 10 grms. of phenol-phthalein were dissolved in 200 c.c. of strong ammonia water (26 per cent.) at the ordinary temperature of the room. The colour formed at

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* Ber. d. chem. Gessell. 11, 1297.

t disappeared in four days without application of heat. I then neutralised with rochloric acid, and obtained the same product as above, which was treated in the e manner.

The three substances so obtained appeared quite similar, and turned red when erately heated in a porcelain dish, melting finally at a higher temperature. They olved in sodium hydroxide solution without colour. When heated to 110° C. with rochloric acid they formed ammonium chloride and phenol-phthalein, as shown by ious tests. They all dissolved in strong nitric acid with a marked yellow colour, ich disappeared on addition of alkali.

It is apparent, therefore, that the phenol-di-imido-phthalein can be formed at or- ary temperature as well as in a sealed tube at 160°, although the preparation of a e amount at the lower temperature would require a much longer time.

The combination between the free alkali and the trace used as indicator in a ctical titration would naturally require but a very short time at the ordinary tem- ature.

The rapidity of liberation of ammonia from an ammonium salt by fixed alkali ends on the temperature, and hence the very slight disturbance of the reaction with mixed solutions in the above experiments carried out at a low temperature. Addi- n of sodium hydroxide seems to aid the reaction with ammonia solution, as shown by eriments in which I added the phenol-phthalein to ammonia solution and then the ium hydroxide from a burette. With half-normal solutions the colour was lost in half hour by mixing 25 c.c. sodium hydroxide, 20 c.c. ammonia, and ·2 c.c. of the indicator. ilar results were obtained with other solutions.

The above experiments show that the phenol-phthalein can be used with monia by taking certain precautions, and they also suggest why the behaviour of this kali was not sooner recognised. By working at a low temperature with a sufficiently ge amount of the indicator, it was shown that fairly sharp results can be obtained. owever, as we have other good indicators, it may never seem necessary to take these ecautions.

After the introduction of the phenol-phthalein as an indicator, some time elapsed fore its extreme sensitiveness was realised, and undoubtedly more was then added to a lution than is now considered necessary. Under these circumstances the difference in haviour between soda and ammonia would scarcely be noticed, and it remained for ter observers, working with less of the reagent, to detect the discrepancy.

I am greatly indebted to my assistant, Mr. Mark Powers, for help in the details of e above experiments.

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

A New Method for the Estimation of Nicotine. Max Popovice (*Journn. de et de chim.*, p. 215).—By the advice of Professor Kossell, the author has set himself study whether the polariscope can be satisfactorily employed for the estimation of nico- e. Pure nicotine is known to be levogyrate, and its rotatory power A_D.—161°·5. In alcoholic or watery solution this rotatory power diminishes. Thus in a mixture of nicotine and water containing 10 per cent. of water, it is only 133°·8. In spite of these variations, M. Popovice has succeeded in successfully estimating the nicotine in tobacco with sufficient accuracy for ordinary purposes. The author employs Kissling's process for extracting the nicotine from the tobacco as follows:—20 to 40 grammes of powdered tobacco, according to quality, are moistened with 10 c.c. of an alcoholic solution of soda in proof spirit of 6 per cent. strength. After standing for some time, the powder is placed in a Soxhlet's apparatus and continuously extracted with ether for three or four hours. The resulting ethereal solution is treated with 10 c.c. of a solution of a sodium phosphomolybdate in nitric acid, which causes the precipitation of the impure alkaloid. When this precipitate has settled, the ethereal solution is carefully poured off, and distilled water is added to the precipitate in the flask until the contents exactly measure 50 c.c. Eight grammes of finely-powdered barium hydrate are then added to liberate the nicotine, and the whole is allowed to stand for several hours with occasional shaking. The contents of the flask are then filtered, and the alkaline filtrate containing the free nicotine is examined by the polariscope. To provide for the variable rotatory power of nicotine already referred to, the author prepared fifteen ethereal solutions of the pure alkaloid, containing proportions comprised between 0·25 and 2·00 grammes of nicotine in 50 c.c. He treated these exactly as above directed, and then examined the alkaline filtrate in a tube two decimetres long. By this means he constructed the following table :—

Nicotine contained in 50 cent. cubes.	Rotation in minutes.	Weight of nicotine corresponding to 1 minute.
2,000	337	
1,875	318	0·00658
1,750	298	0·00625
1,625	278	—
1,500	258	—
1,375	238	—
1,250	217	0·00595
1,225	196	—
1,000	175	—
0·875	154	—
0·750	133	—
0·625	111	0·00569
0·500	89	—
0·375	67	—
0·250	45	—

If, therefore, in an analysis of tobacco we find a rotation somewhere between 337 and 45 minutes, we can obtain the proportion of nicotine by multiplying the number of minutes by the corresponding factor given in the third column. The process is evidently only reliable when exactly followed in every detail. The author has tested it side by side with Kissling's process on a large number of samples, and has found the results to be concordant.

M. S. A. M.

Preparation of Oxygen for Analytical Purposes in a Kipp's Apparatus. A. Baumann (*Zeitschr. f. angew Chemie*, No. 3, 1890).—To prepare oxygen in a Kipp's apparatus, Neumann recommended a mixture of black oxide of manganese, barium dioxide, and plaster, which is made into cubes, and evolves oxygen when decomposed with dilute hydrochloric acid. The gas, however, is not pure, but contains chlorine; so it has to be passed through a wash-bottle containing potash. The author warns against the use of these cubes, as he nearly had a serious accident in his laboratory owing to an explosion taking place when the apparatus, at work, stood too near the light. He also does not recommend Volhard's plan, who decomposes compressed chloride of lime with acid solution of hydrogen peroxide, as the gas is also mixed with much chlorine and carbonic acid. The author now proposes to decompose black oxide of manganese with an acid solution of hydrogen peroxide. A rich ore is chosen and made into small pea-sized lumps, from which any fine powder is removed by means of a sieve. After having put an india-rubber ring in the central bulb of the apparatus, and surrounded it with so much asbestos that no grains of manganese can get into the lower bulb, the central bulb is quite filled with the manganese. Commercial peroxide of hydrogen is now acidified with 150 c.c. of sulphuric acid per litre. The gas is now evolved in the same way as one prepares hydrogen from zinc and sulphuric acid. The oxygen evolved is perfectly pure.

L. de K.

Scheme for the Analysis of Creoline. J. W. Gunning (*Nederl Tydschr v. Pharmacie, etc.*, Nov. 1889).—This disinfectant has become a great favourite on the continent, and is supposed to be harmless. Although chiefly composed of neutral tar oils, these, when the product is diluted with water, get into such an extreme state of division that they possess disinfecting properties. In support of this theory, the fact has been observed that the emulsion soon gets inactive. To get an approximate analysis of the compound, the author operates as follows: The creoline is mixed with petroleum ether of 80° Cent. boiling point. First, the two fluids mix, but on adding more the tar oils dissolve, whilst the resin soap separates, which, after having been washed a few times with petroleum ether, will be found soluble in water. On adding acid the resin separates and may be purified with alcohol, and finally weighed. To identify the resin, its acidity equivalent may be taken. The amount of alkali contained in the article may be done by the usual methods. The acid liquid, from which the resin has separated, may contain a little phenol, which may be recovered by shaking out with ether. The tar oils are recovered by evaporating off the petroleum ether. On cooling, any napthaline will show. Shaking with water will extract any phenol; soda-ley will extract cresol. Bodies of a basic nature may be shaken out with dilute acids, and extracted from the acid solutions by agitating with alkali and ether.

The residual tar oil is dried over calcium chloride, and further identified by its boiling point.

L. de K.

Estimation of Ferric Oxide and Alumina in Native Phosphates. A. Stutzer (*Zeitschr. f. angew Chemie*, No. 2, 1890).—The conventional process (see Analyst, January, 1890) does not yield accurate results, especially as the mixed phosphates of
n and alumina may be contaminated with calcium fluoride or other lime compounds.
sides it is unwarrantable to consider half their weight to be phosphoric anhydride,
re being a sensible difference in the molecular weight of the two compounds. Glaser
certainly made an improvement by introducing s alcohol process, but the author

thinks the best plan is to isolate the ferric and aluminic hydrates free from phosphoric acid or other impurities.

Five grams. of native phosphate are boiled with 50 c.c. of hydrochloric acid of 1·12 sp. gr., and diluted with water up to 500 c.c. 100 c.c. (= 1 gram.) are pipetted off, made alkaline with ammonia, and then slightly acidified with acetic acid. The mixed precipitate of ferric and aluminic phosphates is collected on a filter and once washed with water. After draining, the precipitate is rinsed back into the beaker, and treated with 150 c.c. of the ordinary solution of molybdate for about five minutes at a gentle heat. The phosphoric acid now completely separates, and the iron and alumina remain in the solution, from which they may be thrown down by slight excess of ammonia. As the precipitate carries with it small quantities of molybdic acid, it is advisable to redissolve in hydrochloric acid and once more to precipitate with ammonia.

L. de K.

Detection of the Colouring Matter of Alkanet Root. A. Bujard and A. Klinger (*Zeitschr. f. angew Chemie*, No. 1, 1890).—During an analysis of a quack-medicine, the authors were ordered to investigate the nature of the colouring matter. In the first sample there was not enough colouring matter to make a satisfactory spectroscopic examination, and the yellowish-green colour of the arnica tincture interfered. A second sample of somewhat deeper colour was next examined. Aniline colours were not likely to be present, therefore they had to search for vegetable colours. The tincture was rendered alkaline with ammonia and shaken out with ether, which became of a pale violet colour, which pointed to the presence of orseille. After evaporating the ether, the residue was dissolved in dilute alcohol, but the violet fluid gave, however, a total different spectrum from orseille. Dilute acetic acid turned the liquid red. Soda-ley made it blue. These liquids, the alkaline one more in particular, gave very decided spectra. Those interested in the spectroscopic investigation of similar colouring matters, we refer to the original illustrated article.

In its reaction with ammonia and ether, alkanet behaves like orseille, whilst the pigments of logwood, grape, blackberries, etc., do not colour the ether.

L. de K.

Determination of the Specific Gravity of Heavy Syrups. Ad. Geniesser (*Zeitschr. f. angew Chemie*, No. 2, 1890).—Strange as it may seem, this determination does not always give concordant results, which, the author finds, is caused by the great difficulty of freeing the syrups from air bubbles, even when they are heated a long time on the water-bath. The following process, introduced by the author, gives very concordant, almost scientifically, accurate results:—

The syrup is first passed through a sieve. A sp. gr. bottle of 50 c.c. capacity is now weighed, and after introducing about 30 c.c. of the syrup, it is reweighed after cooling. Suppose, when empty, it weighs 24·6506 grams., with syrup it weighs 79·4220; it will then contain 54·7914 syrup. The bottle is now immersed in an oil-bath or in brine, and this is heated until the syrup begins to boil, which causes all air to be expelled. After cooling to 60° F., a drop of distilled water is put on the top, which will soon break and expel the air bubbles. The bottle is now carefully filled up with water and weighed. Suppose it weighs 89·7660 grams., then 89·7660 – 79·4220 = 10·3240 grams. of water have been added. The bottle holding exactly 50 grams. of water, the syrup occupied the same volume as 50 – 10·324 = 39·676 grams. of water. Its sp. gr. is therefore 54·7914 : 39·676 = 1·38097. L. DE K.

ESTIMATION OF CADMIUM IN ZINC REFUSE. W. MINOR (*Chemiker Zeitung No.* 1, 1890).—The author already published an accurate method in 1889, but this required a four times reprecipitation with hydrogen sulphide. This being very troublesome, the author has now devised another process. A definite quantity of the substance is dissolved in hydrochloric acid, and the cadmium separated from zinc and iron by passing a current of hydrogen sulphide. The precipitate, which always contains some coprecipitated zinc, is washed with hot water and dissolved in dilute hydrochloric acid. This solution is heated to boiling, and poured into a boiling solution of sodium hydrate. The precipitated cadmium oxide is filtered off and completely freed from zinc by washing with a 1 per cent. solution of caustic soda. The alkali is now perfectly removed by means of boiling water, and the filter is then dried. To prevent loss by reduction, the filter is ignited in a Rose's crucible, in a current of pure oxygen. The weighed cadmium oxide is then calculated to metal. If iron should be absent, the precipitation with hydrogen sulphide may be omitted, and the cadmium solution at once poured into the caustic soda.

To estimate zinc and cadmium in native zinc sulphide, the author recommends the following process. The ore is dissolved in the usual way, and then mixed with excess of ammonia which throws down the iron, and redissolves the zinc and cadmium. This ammoniacal solution is now nearly neutralised and poured into excess of boiling caustic soda, which throws down the cadmium and keeps up the zinc. The cadmium oxide is treated as described, whilst the zinc solution after neutralising the greater part of the alkali with hydrochloric acid, may be titrated with standardised sodium sulphide.

L. DE K.

FAT TITRATION. MAX GRÖGER (*Zeitschr f. angew Chemie, No.* 3).—In many analyses, such as the iodine absorption for fats, the estimation of carbolic acid with bromine, etc., etc., it is a well-known fact that unless a large excess of the reagent has been added, the results are untrustworthy. The author now finds the same sort of thing with the Koetstorfer process for fats. No result must be trusted unless the produced soap requires at least 5 c.c. of semi-normal hydrochloric acid for neutralisation.

L. DE K.

A RAPID METHOD FOR PHOSPHORUS IN IRON AND STEEL. BY G. L. NORRIS (*Journ. Frank. Inst., Jan.*, 1890).—Five grams. of pig-iron or steel are treated in a twelve-ounce covered beaker, with 90 c.c. of 1·135 sp. gr. nitric acid for steels, 120 c.c. for pig-irons. The beaker is heated on an iron plate until all action ceases and solution begins to boil. To the boiling solution 20 c.c. of a solution of potassium permanganate, eight grams. to the liter, are added. The solution is then boiled a few minutes, to be sure all the permanganate is used up. A precipitate of manganese peroxide must come down on boiling to be sure that all the phosphorous is oxidised. After boiling with permanganate, a small piece of tartaric acid is added, and the solution boiled a minute or two longer, until all the manganese peroxide is dissolved. The solution is then poured in a 500 c.c. globe flask and 10 c.c. of 1·40 sp. gr. nitric acid added. The solution in the flask should then be about 90 c.c. in bulk. In case of pig-iron the solution is made up to 100 c.c. with water, the graphite filtered off, and 80 c.c. of the filtrate drawn off into a flask, and then 10 c.c. of 1·40 sp. gr. nitric acid added. The solution is then heated to 90° C., and 80 c.c. of ammonium molybdate solution added, the flask stoppered, wrapped in a cloth, and shaken hard for five minutes. The molybdate solution is made by dissolving 100 grams. molybdic acid in 400 c.c. strong ammonia and then mixing with 1,200 c.c. of 1·20 sp. gr. nitric acid. From this point on the method is the same as Emmerton's, reduction of the yellow precipitate with zinc and titration with a standardised permanganate of potassium solution. For high phosphorus pig-iron only 1·25 grams. are used and treated with 60 c.c. of 1·135 sp. gr. nitric acid. The solution is made up to 100 c.c., filtered, 80 c.c. of the filtrate drawn off, and 10 c.c. of 1·40 sp. gr. nitric acid added.

The only speed determination the author has made was on a steel, and thirty-six minutes covered everything, weighing and all. The test analyses are very satisfactory.

M. S. A. M.

LAW NOTES.

THE LEGAL PLACE OF DELIVERY FOR COUNTRY MILK.

THIS was an appeal from the decision of the borough justices convicting Messrs. Lush, farmers, near Basingstoke, of sending adulterated milk to Mr. Lumsden, dairyman, Reading.

Mr. David (instructed by Mr. Sidney Brain) appeared for the appellants, and Mr. A. H. Spokes (instructed by Mr. F. Stevens, solicitor, Assistant Town Clerk) for the Reading Sanitary Authority.

Mr. Spokes opened the case for the Sanitary Authority. He stated that the Legislature felt that the Sale of Food and Drugs Act, 1875, was not sufficient protection to the retailer of milk in towns, who knew nothing about the milk which he received from some farmer in the country, and an Act of Parliament was therefore passed in 1879 to remedy that state of things. Any inspector of nuisances or officer having charge of the execution of the Act could procure a sample at the place of delivery and take proceedings, where the article was sold or actually delivered to the purchaser, which was in this case at Reading Station. He quoted a decision of Mr. Justice Field, that it was the intention of the Act to hit the person who sent the milk from the country. The learned counsel then stated that the contract was made in March, 1889, and the milk was delivered at Basingstoke till August 11th, when part of it was sent to Reading. On September 11th directions were given to send the whole of the milk to Reading. His learned friend was going to rely on the technical rule that the railway company were agents for the consignee. This case was exactly like the case of the delivery of milk from Coventry to Euston Station, in which nobody suggested that the delivery was at Coventry, and not at Euston Station, and the Court dealt with the case as if the milk were delivered at Euston Station.

Mr. Sinclair Campbell Lumsden, milkseller, Minster Street, Reading, and Vine Dairy, Basingstoke, said that in March, 1889, he made a verbal contract with Mr. Thomas William Lush and Mr. Duncan Lush, farmers, Basingstoke, to deliver milk at two prices to Vine Street, Basingstoke, for twelve months. From September 11th the milk was sent to Reading Station. On September 13th witness sent a telegram to his manager to send Lush's milk that day, but no more.

Edwin Perkins, manager to Mr. Lumsden, at Basingstoke, proved that the delivery of the milk was first at Basingstoke. He sent a note by Mr. Lush's man, North, to send the milk to Reading Station. He afterwards had a conversation with one of the Messrs. Lush, and after August 11th some of the milk was sent to Reading, and the youngest Mr. Lush sometimes delivered it at the Basingstoke Station. Witness supplied the labels to put on the churns by Messrs. Lush or their men. On September 13th witness received the telegram alluded to, and sent it to Mr. Lush after their men had been to the Basingstoke railway station with the milk.

Cross-examined: The answer of Mr. Thomas Lush first was that they were willing to deliver anywhere in Basingstoke. They were willing afterwards to take the milk to Basingstoke Station to go to Reading Station. He could not refer to any specific interview after that, but he knew there were several. He never took any milk out of the churns at Basingstoke Station.

Re-examined: They never objected to take the milk to Basingstoke Station.

David North, in the employ of Messrs. Lush, said that on September 13th he took two churns from Messrs. Lush's farm to Basingstoke Station. Mr. Thomas Lush put the labels on the churns at the farm. Witness took the churns out of the cart and put them on the platform, told the porter, and he booked them. Witness saw the milk put into the train by the porter. Neither Mr. Perkins, nor Mr. Lumsden nor any of his men were at the station. Witness took the milk to the Vine Dairy and to the station at Basingstoke when it was required. Witness met Mr. Perkins about half a mile from the station, and received from him the telegram referred to.

Cross-examined: The milk was under his sole charge at the farm, and up to the time he got to the station. He did not put anything in the milk. The churns were fastened down, but not locked. They could have been opened by anyone, but it would have taken some time in doing it.

Alfred Vicars, parcels' porter at the Basingstoke Station, corroborated the last witness, and said the churns were near the office door, and no one could have put anything into them without being seen.

Mr. W. H. Roberton, Inspector of Nuisances at Reading, proved taking a sample of milk from one of the churns when they arrived at Reading, and submitting the sample to Dr. Ashby, the public analyst.

Mr. Wright, manager of Mr. Lumsden's Reading depôt, gave corroborative evidence, and said he took the milk away from Reading Station.

Dr. Ashby proved that the milk was adulterated with not less than eighteen per cent. of added water.

Cross-examined: He took a standard which would provide against all contingencies, such as poor cattle and poor land. The milk was not like skim milk or natural milk. It was like milk with water added to it.

Mr. David submitted that the place of delivery was Basingstoke and not Reading; that the contract for delivery was at Basingstoke, and that there had never been any variation of the contract; and that the delivery took place and was completed at Basingstoke. The Inspector should have given one of the samples to Mr. Lush.

Mr. Spokes replied on the case, and submitted that a sample could not be taken at Basingstoke Station.

The Recorder said he was clearly of opinion that the conviction should be supported, because it properly came within the terms of the Act. He confirmed the conviction, and ordered the appellants to pay the costs of the appeal.

Mr. David asked for a case upon the point of delivery.

The Recorder said he would grant one if he thought there was any doubt upon the point. He had considered that point, and could not think there was a shadow of a doubt, and being of that opinion, he should stultify himself if he granted a case.

GUILTY KNOWLEDGE OF SKIMMING NOT NECESSARY FOR CONVICTION.

This was a case stated by the Metropolitan Police Magistrate at Dalston, and it raised the question whether it is necessary, in order to convict a vendor of milk under section 9 of the Food and Drugs Act, 1875, to show that he knew the milk had been altered.

Mr. Austin appeared for the appellant, the Inspector of Nuisances for St. Mary's, Islington, against the dismissal of the information by the Court below. On July 25th the respondent was summoned for having sold, on June 24th, 1889, an "article of food, to wit, milk, in its altered state without notice or making disclosure of the alteration, from which 28 per cent. of its original fat had been abstracted, so as to affect injuriously its quality, substance, or nature, contrary to the provision of the Sale of Food and Drugs Act, 1875, 38 and 39 Vict., c. 63, and the Sale of Food and Drugs Amending Act, 1879, 42 and 43 Vict., c. 30." It was proved that the appellant bought a pint of milk at the shop of the respondent. It was duly divided into three parts, one of which was sent to the public analyst. The certificate of the public analyst stated that 28 per cent. of fat had been abstracted, and the result of the analysis showed 1·47 per cent. of fat was all that was left. Except the certificate, no evidence was given that the milk had been altered, and the defendant gave evidence that he had no knowledge of any such alteration, and his daughter, who sold the milk and managed the shop, also denied all knowledge of any alteration. The magistrate held that, as the 9th section contemplated disclosure by the seller of the alteration of any article, it was necessary to prove knowledge of the alteration, and in the absence of such proof dismissed the summons. Mr. Austin cited the cases of "Betts *v.* Armstead" (20 Q.B.D., 771) to show that it was an offence under section 6 to sell an article not of the quality demanded by the purchaser, although the vendor did not know that it was not of the quality demanded. "Cox *v.* James" (L.R. 7, Q.B. 135) was a decision under a different Act, and was not an authority against this view. "Cundy *v.* Lecocq" (13 Q.B.D., 207) showed that a *mens rea* was not essential when the act was absolutely prohibited. "Lane *v.* Collins" (14 Q.B.D., 193) was also referred to. 38 and 39 Vict., c. 63, sec. 9, is as follows:—

"No person shall, with the intent that the same may be sold in its altered state without notice, abstract from an article of food any part of it so as to affect injuriously its quality, substance, or nature, and no person shall sell any article so altered without making disclosure of the alteration, under a penalty in each case not exceeding £20."

By section 25 it is provided:—

"If the defendant in any prosecution under this Act prove to the satisfaction of the Justices or Court that he had purchased the article in question as the same in nature, substance, and quality as that demanded of him by the prosecutor, and with a written warranty to that effect, that he had no reason to believe, at the time when he sold it, that the article was otherwise, and that he sold it in the same state as when he purchased it, he shall be discharged from the prosecution."

The respondent did not appear.

Mr. Justice Grantham regretted that no counsel had appeared on the side of the respondent, but he felt no doubt that the appeal must be allowed. In 1875 the Legislature incorporated the old Acts about adulteration of foods and drugs. The first clauses of the Act 38 and 39 Vict., c. 63, dealt with the mixing of injurious ingredients with the articles sold. Then section 5 provided that no person should be liable to be convicted under the previous two sections if he could show that he did not know of the article having been so mixed, and that he could not with reasonable diligence have obtained that knowledge. Section 6 prohibited the sale of food and drugs not of the quality demanded. With a full knowledge of the fact that ignorance of the adulteration is stated to be a ground of exemption, section 9 was passed without any such exemption. That section dealt with two classes of persons, first with those who abstracted from an article of food, and secondly with those who sold the goods so altered. It would have been useless merely to forbid the alteration of the goods if the sale of the goods so altered were not also prohibited; for, if not, the retailer would sell it and the public would buy. The 25th section enabled a retailer to defend himself by obtaining a written warranty from the original vendor. He might hand this to the inspector of nuisances when charged with the offence. If a person did not take the trouble to get a warranty he must be held liable for breaking the law when he sold that which was expressly forbidden by the statute to be sold.

Mr. Justice Charles was of the same opinion.

LEGAL EFFECT OF A NOTICE OF DILUTION UNSEEN BY THE PURCHASER.

This was a case stated by justices, and raised a question under the Sale of Food and Drugs Acts. The appellant, an inspector under the above Acts, sent two of his assistants to the respondent's public-house, and they went into the club-room, owing to the bar and kitchen being full, and asked for and received half a pint of whisky. The respondent was thereupon informed that the whisky was purchased for analysis, and it was divided into three parts, in accordance with the statute. Upon analysis it was found that the whisky was diluted with water to the extent of 37½ per cent. under proof. It was proved that a notice was placed in a conspicuous place in the bar and in the kitchen that all spirits sold at that establishment were diluted with water according to price. There was no notice to this effect in the club-room. The interior of the bar was visible to a person going into the club-room. The justices found as a fact that the appellant's assistants did not see or know of any notice being posted in any part of the house. The respondent was summoned before the justices under section 6 of the Sale of Food and Drugs Act, 1875 (38 and 39 Vict., c 63), for selling to the prejudice of the purchaser an article of food, to wit, whisky, which was not of the nature, substance, and quality of the article demanded. Section 6 of the Sale of Food and Drugs Act, 1879 (42 and 43 Vict., c. 30), allows whisky to be diluted with water to the extent of 25 per cent under proof. The justices refused to convict, but stated a case for the opinion of the Court as to whether they ought to have convicted.

Mr. H. D. Green, Q.C., appeared for the appellant, and referred to "Sandys *v.* Small" (3 Q.B.D., 449); "Hoyle *v.* Hitchman" (4 Q.B.D., 233); "Gage *v.* Elsey" (10 Q.B.D., 518). He contended that the notice not having been been brought to the knowledge of the purchasers, the sale was to their prejudice.

The respondent did not appear.

The Court remitted the case to the justices.

Mr. Justice Mathew said that it was manifest the respondent was carrying on business in the ordinary way, and had no intention of deceiving these purchasers. It was extremely curious that the purchasers did not go into the ordinary parts of the house, where they must have seen the notice. They, however, found their way into the club-room, where they were supplied with the spirits. Under these peculiar circumstances he (the learned Judge) was disposed to be extremely critical as to the findings in the case, and in his opinion it was incumbent upon the complainants to satisfy the justices that they did not know that the spirits sold in the house were diluted. There was no finding upon that point in the case. The only finding was that the complainants did not see the notice. The Court could not, therefore, satisfactorily dispose of the case without sending it back to the justices to find as a fact whether the purchasers knew that spirits sold at the respondent's house were diluted. If the justices found in the affirmative there must be no conviction; if in the negative, then the respondent should be convicted.

Lord Justice Fry said that he had doubts whether the burden of showing knowledge in the purchasers was not upon the respondent, but he was strongly inclined to think that the respondent could discharge that burden. Under the peculiar circumstances the case must go back to the justices.

REVIEW.

NOTES ON QUALITATIVE ANALYSIS, ARRANGED FOR THE USE OF THE STUDENTS OF THE RENSSELAER POLYTECHNIC INSTITUTE. By W. P. MASON, Professor of Analytical Chemistry. 2nd Edition. Troy, N.Y.: Nims and Knight.

THIS is a well-printed little book, possessing no very special features except that, like many works of its kind, it does not contain any attempt at an acid course worth a name. Here and there we come across a really useful note, such as one showing the student the things that must not be evaporated in a platinum dish. Over here we have many such booklets, and we do not think that there will be any room for this one in the British market. A point is made of giving always a reference to larger books for full information on matters beyond the scope of the present one, which is a commendable feature.

THE ANALYST.

APRIL, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

AN ordinary meeting of the Society was held at Burlington House on Wednesday, the 12th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

A ballot was taken for new members, when the following were declared elected:—R. Hellon, Public Analyst for Cumberland; J. B. Harrison, Government Analyst, British Guiana.

Mr. H. D. Richmond, of Cairo, was proposed as a member.

The following papers were read and discussed:—

"On the Analysis of Disinfecting Powders containing Commercial Carbolic and Sulphurous Acids."—By Dr. MUTER.

"On the Chromate Test for Lead in Water."—By SIDNEY HARVEY.

"On the determination of Rosin in Soap."—By ROWLAND WILLIAMS.

The next meeting of the Society will be held at Burlington House on Wednesday, the 9th inst.

ON THE SOLUBILITY OF PHOSPHATE OF ALUMINA IN ACETIC ACID WITH SPECIAL REFERENCE TO THE ESTIMATION OF ALUMINA IN FLOUR, BREAD, ETC.

BY W. C. YOUNG, F.I.C., F.C.S.

(Read at Meeting, February, 1890.)

IN previous communications to the Society I have called attention to the solubility of phosphate of alumina in acetic acid and its effect upon the "logwood" test. I propose in this paper to show to what extent this solubility affects the processes in use for the determination of alum in bread, etc., where the phosphate of alumina is precipitated in the presence of free acetic acid, and the influence exerted by the acetate of ammonium and other compounds present in solution and by proceeding in a variety of ways.

The solutions used in the experiments to be described were of the following strengths, viz.:—Alum, 100 c.c. = ·1 gram. potash alum, phosphate of sodium, 100 c.c. = 20 grams. acetate of ammonium, 100 c.c. = 20 grams. acetic acid, commercial pure.

The experiments were made in the following manner: 100 c.c. of alum solution taken in each case (the quantity of alumina present representing the amount that would be obtained from 100 grams. of bread containing about 20 grs. of alum in 4 lbs.)

No. 1. 5 c.c. acetic acid, 2 c.c. phosphate of sodium solution, and 5 c.c. acetate of ammonium solution added, boiled and then filtered.

No. 2. 5 c.c. acetic acid and 2 c.c. phosphate of sodium solution added, boiled, then 5 c.c. acetate of ammonium solution added, again boiled, and then filtered.

No. 3. 5 c.c. acetic acid, 2 c.c. phosphate of sodium solution, and 5 c.c. acetate of ammonium solution added, set aside in the cold and filtered as soon as the precipitate settled.

No. 4. 5 c.c. acetic acid, 2 c.c. phosphate of sodium solution, and 5 c.c. acetate of of ammonium solution added, set aside over-night, then filtered.

No. 5. 5 c.c. acetic acid, 2 c.c. phosphate of sodium solution, and 5 c.c. acetate of ammonium solution added, boiled, and set aside over-night, then filtered.

The various solutions were added in the order given above.

In every case the liquid remained quite clear in the cold until the acetate of ammonium solution was added, when a precipitate commenced to form. In the case of experiment No. 2, a precipitate formed on boiling, which, after addition of acetate ammonium, became very dense, much more opaque and apparently larger in bulk than any of the others.

The results obtained, stated in grams., are as follows:—

	A.	B.	C.	Mean.	
1.	·0230	·0230	·0210	·0223	grammes.
2.	·0250	·0245	·0250	·0248	„
3.	·0218	·0220	·0205	·02143	„
4.	·0226	·0225	·0215	·0222	„
5.	·0226	·0225	·0230	·0227	„
		Theory gives	·0258.		„

It will be seen from these figures that in every case the result was below theory, the nearest approach being in the case of experiment No. 2.

It is curious that in No. 2 the character of the precipitate obtained differed from all the others and was larger in amount, as the only difference between it and No. 1 was that the solution was boiled after the addition of the phosphate of sodium, and again after adding the acetate of ammonium, whereas in No. 1 all the solutions were added before boiling. The lowest result obtained was in No. 3, as was to be expected.

On comparing No. 1 and No. 5 it will be seen that there is very little difference, showing that after precipitation by boiling the phosphate of alumina does not redissolve by long contact with acetic acid of the strength employed in these experiments. The increase in the precipitate by boiling is shown on comparing No. 1 with No. 3. The influence of the acetate of ammonium being so very marked I repeated experiments 1, 3, and 4, using in each case 1 c.c. of acetate of ammonium solution, or a fifth of the quantity used before, when I got the following results:—

1. ·0185 3. No precipitate. 4. ·0108 grms.

Experiment No. 2 was also repeated, using 15 c.c. of acetic acid and no acetate of ammonium, the result being ·0165 gram.

No. 1 was the result obtained by filtering immediately after boiling, No. 3 in the cold, and No. 4 after boiling and setting aside over-night. No. 2 shows the degree of solubility of phosphate of alumina (precipitated from a boiling solution), with a large excess of acetic acid in the absence of acetate of ammonium. These results show very clearly that the quantity of acetate of ammonia present has a great influence on the solubibity of phosphate of alumina in acetic acid.

In order to ascertain if the quantity of phosphate of alumina dissolved increased with the proportion of alum present, I repeated the five experiments, using a ·2 per cent. solution of alum, with the following results:—

	A.	B.	C.	Mean.	
1.	·0480	·0475	·0470	·0475	grammes.
2.	·0505	·0500	·0500	·0502	,,
3.	·0445	·0440	·0435	·0440	,,
4.	·0450	·0460	·0460	·0457	,,
5.	·0470	·0460	·0450	·0460	,,
		Theory gives	·0516		,,

On comparing these figures with the first series it will be seen that the loss in every case is slightly increased.

The following experiments, in which 15 c.c., instead of 5 c.c., as in the former were used, show the effect of a large excess of acetic acid.

THE ANALYSIS OF CARBOLIC AND SULPHUROUS DISINFECTING POWDERS.

By John Muter, Ph.D., F.R.S.E.

Read at Meeting, March, 1890.

When our Secretary pressed me to read a paper to fill up an apparent void at the February meeting, I thought that it would not be uninteresting to initiate a discussion on this subject, which is daily becoming more and more important to public analysts. Local authorities are waking up to the use of the analyst as a protector of their interests against the deceptions too often practised by unscrupulous persons, and therefore it is well that some agreement should be come to among ourselves as to the best process to employ, and also as to the best form of tender to be recommended for use by such bodies in obtaining contracts. As the latter has become really a very burning question, both to the purchasers and the trade, I propose to take it first.

The common, loose form of contract is for "carbolic acid powder containing 10 (or haps 15) per cent. of liquid carbolic acid at so much per ton," to which is frequently led the stipulation that "the base shall not be lime." If the words of such contract to be interpreted in a strictly chemical sense, viz: that the powder shall contain to 15 per cent. of absolute phenol, the whole thing becomes a farce; because no such powder could be produced under a cost that would be absolutely prohibitory. All chemists know that the article commonly sold as "liquid carbolic acid" is not absolute phenol rendered liquid by the addition of 5 to 10 per cent of water, but consists of the higher phenols of coal-tar (chiefly cresol) from which the phenol itself has been nearly absolutely removed. Cresol has been held to be just as good an antiseptic as phenol, provided that it be in a free state, and especially not combined with lime. Its comparatively cheap cost, moreover, has caused it to be almost entirely employed in the manufacture of carbolic powders. Why, therefore, should this fact not be openly recognised, and all sorts of difficulties saved at once to the manufacturers, the medical officers of health, and the analyst. An analyst dealing with such a powder, and most probably employing the process originally described in the ANALYST for October, 1887, or some modification thereof, naturally reports that the powder contains so much per cent. of tar phenols (chiefly cresol); this report being laid before the authorities, some one immediately starts up and exclaims, "This is not what we want; we contracted for carbolic acid," and then the difficulty begins. An appeal is made to the medical officer, and he, answering on the spur of the moment, without an opportunity of conferring with the analyst, may simply increase the difficulty. That this is no fancy picture is evidenced by the fact that it has actually occurred in many cases, and I have at the moment in my mind an instance in which a firm, having taken such a contract, have lately been threatened that they will be compelled to supply a powder containing 10 per cent. of pure, absolute phenol, the medical officer declaring that he contracted for carbolic acid, by which he means phenol, and nothing else. In some districts the powder is also required to contain 5 per cent. of sulphurous acid without the use of the word "available," and some day we will probably meet with the *reductio ad absurdum* in the shape of a demand by a medical officer for such a powder containing 5 per cent. of free sulphurous acid—a chemical absurdity. I would therefore submit to you for consideration the advisability of impressing your Boards and the manufacturers with the necessity of ceasing to employ trade misnomers, and of contracting for a powder, the base of which shall be gypsum or siliceous matter, and which shall contain a certain per centage of uncombined tar phenols (chiefly cresol), and also, if desired, 5 per cent. of *available* sulphurous acid. I feel satisfied that in our progressive times nothing is lost, either to manufacturers or to consumers, by calling a spade a spade.

Turning now to the analytical question, I will proceed to make a few short remarks as to the basis of what will, I hope, prove to be an interesting discussion. I still estimate the phenols in carbolic powders by the process described by Mr. De Koningh and myself in the ANALYST for October, 1887, nor, despite the criticisms which subsequently appeared by Mr. Williams in the *Journal of Chemical Industry*, have I seen any necessity to alter it. The only modification I have come to in practise consists in employing 150 c.c. of 10 per cent solution of sodium hydrate instead of 200 c.c. of a 5 per cent. solution. It must also be remembered by all persons using the process that the acids, when measured over the brine, contain about 5 per cent. of water; because we have proved by experiment that commercial anhydrous cresol shaken up with brine absorbs this amount.

Speaking of commercial cresylic acid, our salt test (described in the same number of e ANALYST) is a good and ready test to enable one to rapidly decide whether a given mple of cresol really contains water or not. Really anhydrous cresol when shaken up ith three volumes of brine, gives an increase in volume of about 5 per cent. If the esol is watery, it will either not increase in bulk at all, or it will decrease slightly. he test is of course only a safeguard against gross frauds, and for the really accurate timation, I use the distillation process, which, when not carried too far, will enable to read off exactly the c.c.'s of water. The amount of cresol dissolved in the water is so ight that it may be disregarded; but, on the other hand, if any appreciable amount of id has come over, about one-tenth of its volume must be added to the water.

The ordinary cresol of commerce usually contains some napthalene, and a rapid ethod of getting at a fairly accurate idea of its amount is a desideratum. This presence naphtalene is often preferred by the purchasers who have the idea that, if "commercial carbolic acid" becomes milky when put into water, it shows its strength. The ocess I have described is as follows:—

50 c.c. of the acid are shaken up with 200 c.c. of 10 per cent. solution of sodium drate, when the acids readily dissolve, leaving the naphtalene floating on the top. The ttom liquid layer is run off, a washing of 5 per cent. solution of sodium hydrate is put , and the whole is rapidly filtered through a quick filter and allowed to drain. The llected naphtalene is then rinsed off the filter into a small beaker with water, and is en once more collected on a pair of counterbalanced filters in the usual manner. After aining, the filters are removed from the funnel and dried as far as possible between otting paper by judicious pressure. The filters are separated, and the inner one and contents are weighed, using the outer one as a tare. The amount of moisture held by e two filters is sufficiently alike to enable us to get a very fair approximation to the ue amount of naphtalene, quite nearly enough for ordinary purposes.

Taking now the analysis of sulphurous powders, the process I use for the estimaon of the available sulphurous acid is as follows:—

Two grams. of a fair sample of the powder are placed upon a small filter, and ercolated with anhydrous ether until the phenols and tarry matters are removed, hich is readily and quickly attained. 50 c.c. of decinormal iodine solution are then laced in a bottle, and as soon as the ether has dried off from the powder, the conents of the filter are dropped into the bottle. The bottle is allowed to stand for halfn-hour (being shaken at intervals,) and, finally, the contents of the bottle are titrated ack with decinormal "hypo" in the usual manner. The amount of iodine destroyed f course represents the available sulphurous acid in the two grams. of powder started ith, and, therefore, it is only needful to multiply the number of c.c. of iodine used by 0032. This method I find to work well with every sort of powder in the market, proided its base be not lime. To get a really satisfactory process in the presence of a lime ase is very troublesome, and I cannot say that I have yet met with one that is at once apid and good. If any member of the meeting knows one, he would be doing us all favour to give it to us.

We come now to the important question of "reversion." Sulphurous powders xidise with great rapidity, and a perfectly honestly-manufactured one may, if badly ept for even a very short time, show, on analysis, a much smaller portion of available sulphurous acid than it ought to do. It is, therefore, the duty of an analyst desirous of holding the scales of justice fairly, to try, if possible, to ascertain the amount of such reversion. With a powder made on a gypsum base and with calcium bisulphite I am not aware that this is attainable; but many of the leading makers recognising this, are so constructing their powders as to render it possible. Some use a purely siliceous base, containing only a neglectable quantity of sulphate, while others use a gypsum base, but put in the sulphite in the form of a specially-manufactured sodium salt. It would, at first sight, appear that the analysis of such a powder is a very simple matter, but this is not so. The whole thing is complicated by the fact that the moment water is put upon the powder, a double decomposition occurs, and we get insoluble calcium sulphite and sodium sulphate; and we find, in practice, nearly the whole of the sulphurous acid in the insoluble residue. To get at the reversion in such powders I have devised the following method :—

20 grams. of the powder are put into a bottle, and 200 c.c. of water are measured in with a pipette. After an occasional shake, the whole is allowed to settle, and some of it is poured off or filtered through a dry filter. The decomposition above alluded to often occurs at this stage of the process; but, as will be seen, the latter is not thereby affected. 20 c.c. of the liquid (=2 grams. of the sample), are mixed with an excess of bromine, and filtered from any bromo-cresol remaining undissolved. It is then treated with an excess of barium chloride, and the total sulphuric acid is weighed as barium sulphate. As, however, a part of this is due to dissolved calcium sulphate, another 20 c.c. are precipitated with ammonium oxalate, and the lime weighed as carbonate. The calcium thus found is now calculated to its equivalent amount of sulphuric acid, and this is deducted from the total. The remainder is still further reduced by deducting an amount of sulphuric acid equivalent to the sulphurous acid previously found by the iodine method, and the remainder represents the reverted sulphurous acid, and is calculated as such.

If the powder be made with a siliceous base, it may be more convenient for those who, only doing the analysis now and then, prefer a gravimetric method, to proceed as follows :—

Drop two grams. of the powder into a beaker containing an excess of bromine water, and while this is acting, take another 2 grams., put it into a dish, and moisten with fuming hydrochloric acid (free from chlorine). Now evaporate to dryness, redissolve, and then throw down the sulphate with barium chloride in both parts. The difference between the two results calculated to sulphurous acid will give the amount of that body present in the 2 grams. of sample taken. If the base be all but free from sulphates, we thus get a fair idea of the probable reversion at the same time.

Discussion.

Mr. Blount said that a superstitious value was generally attached to the disinfecting powers of the lower tar acids. In the specification for creosote used by the Crown Agents for the Colonies and in vogue among engineers, it was laid down that the creosote should yield not less than 10 per cent. of tar acids, of which at least half "is to be carbolic acid, distilling at a temperature not exceeding 450° F." (233° C.). Having regard to the ambiguity of the term "carbolic acid," and the risk of its being looked upon as equivalent to *phenol*, he preferred in reporting upon samples examined under this specification to use the phrase "tar acids distilling below 450° F." as more accurate. It sometimes happened, however, that a client stickled for "carbolic acid," in which case he was accustomed to write the words in quotation marks, and indicate that the specification was responsible for them.

As to the question of extracting the tar acids, most of the creosotes he saw were of good quality, and yielded more than 10 per cent. of tar acids when the distillate below 610° F. was extracted with caustic soda, thus complying with the specification; but such samples as only just contained the permitted minimum of tar acids, and from which it was therefore important in judging them to get out every trace, were distilled to the point of pitching and then extracted.

Mr. Cassal said that in the analysis of creosotes he had found that unless the distillation was carried to the point of pitching, the whole of the "tar acids" were not got out. He had found, further, that the passing or condemnation of a sample might depend upon the difference made by carrying the distillation to this point. Particular specifications had to be followed in which, as a rule, certain definite percentages of "crude tar acids" were mentioned. In one with which he had frequently to deal, the amount of "crude tar acids" was fixed at 8 per cent. for creosote intended for the treatment of timber, and this could certainly not be regarded as a high standard to take. Specifications for the supply of carbolic powders and liquids were, like those in use for creosote, of a very variable character. There were, in fact, all sorts of specifications and all sorts of rules laid down; it was very desirable to find out according to what authorities they had been prepared, and if possible to arrive at some sort of understanding upon the whole matter, which was at present in a state of much confusion.

He desired to mention further, firstly, that he had found it necessary in determining the tar acids to wash repeatedly with the soda solution in successive separate quantities; and secondly, after the separation of the acids to allow them to stand over the salt and acid solution for at least twelve hours. When these steps were not taken, the results were always too low, and sufficiently so to make a considerable difference.

Referring to the use of the term "Carbolic Acid," Mr. Cassal said that it could not be taken as synonymous with Absolute Phenol. The term "Carbolic Acid" had always been applied to a mixture of phenol bodies; this was the recognised trade meaning of the term, and to assert that it meant absolute phenol was to take an improper advantage of the existing confusion in chemical nomenclature. He agreed with Dr. Hunter as to the unnecessary difficulties introduced by such a proceeding. If medical men and sanitary authorities, for reasons no doubt best known to themselves, but which they would find it difficult to specify, required absolute phenol to be supplied to them, they ought to use the proper chemical term, and they would then be justified in calling upon the analyst to decide whether they had been supplied with what they had asked for.

Mr. Adams said that phenol was much more active as a disinfectant than as a deodorant, and there was a great difference between the two. As a true disinfectant, he thought the medical profession generally held phenol as the best of all the tar acids. Speaking for himself, he had not much confidence in any; he was quite positive that, in the ordinary way in which they were used, they exert no power as bacteriacides. He had several very interesting facts in connection with that point, and one was so clear that it was worth relating there. Many years ago he had some papers brought to his house from the secretary to his hospital. He knew that some of the secretary's children had just died of violent scarlet fever, and was consequently rather anxious about his own children; he therefore drenched the papers with a strong solution of carbolic acid, and then placed them in a window at the top of his house, exposed to the sun and out of reach, as he hoped, of his children. He was working one day at a lathe in this room, when one of his little children got hold of some of the papers. He took her at once and washed her hands, but after three days that child had the scarlet fever. A more distinct proof of the source of infection than that could hardly be. He did not pretend that the whole of the papers were drenched through and through, but all the outside was, and they were exposed for months in the sunlight. Personally, therefore, he had very little belief in the use of carbolic acid as a bacteriacide; but still he did consider it was a valuable disinfectant. It had an inhibitory power over the germs, and for the time suspended their activity, but as soon as the carbolic acid had evaporated, unless it was sufficiently strong, it left the bacteria as active as ever. He could mention many other instances which had caused him to come to the conclusion that coal tar acids were useless for the positive destruction of disease germs. He had a different opinion about sulphurous acid, but the ordinary way of burning a few ounces of sulphur in a large room was not enough. A pound to a thousand cubic feet of space was required.

ON THE CHROMATE TEST FOR LEAD IN WATER.

By Sidney Harvey, F.C.S., F.I.C.

Read at Meeting, March, 1890.

As my paper "On the detection of lead in potable Waters by means of Potassium Bichromate," published in the Analyst for 1881 (vol. 6, pp. 146), has apparently escaped the notice of several chemists, and as I have since had abundant opportunities of testing the extreme delicacy of the chromate reaction, I feel justified in again drawing the attention of analysts to the subject, referring for full details to my previous paper.

The H_2S test for metallic impregnation is of course a most essential one, but it is not always safe to ascribe the colour thereby struck to lead thus ignoring the possible presence of copper and tin, and it is most desirable before proceeding to quantitative results to be sure of the presence of the very metal suspected. For the purpose of proving the presence of lead nothing in my experience exceeds the delicacy of the chromate reaction, which is performed as follows :—

Half a litre of the water in question, if it can be spared (otherwise quarter of a litre will do), is placed in a conical precipitating jar (Phillips's) about two grains of $K_2Cr_2O_7$ are added, and dissolved by agitation. The mixture is set alongside another jar containing "lead-free" water treated in a similar manner. It is assumed that the water is quite clear, if not it must be carefully filtered to render it so; the addition of any acid is objectionable, and previous concentration is unneccessary and even injurious. The use of the bichromate in *crystals* is also essential.

Water containing as little as one fiftieth of a grain per gallon of lead will, when thus treated, become sensibly turbid in about fifteen minutes, and the turbidity is rendered the more apparent by contrast with the jar alongside. In about twelve hours, if undisturbed, the precipitate will have completely settled, and the fluid may be poured off to the *last drop*, leaving the bottom of the jar coated with the $PbCrO_4$, which latter may be rendered much more distinct by mixing it with a few c.c.'s of distilled water, and allowing the same once more to settle in a narrow flat bottom tube. Of course, in the case of waters containing larger amounts of lead than the above, the reaction is much more prompt and decisive.

No other metal likely to be present in water will give a similar reaction, and I am inclined to think that this test for lead is amply sufficient for all practical purposes. One fiftieth of a grain per gallon of lead is equivalent to one part in three and a half millions or to one part of $PbCrO_4$ in two and a quarter millions, thus demonstrating the remarkable insolubility of chromate of lead.

(*Conclusion of the Society's Proceedings.*)

DETECTION AND QUANTITATIVE ESTIMATION OF INORGANIC AND ORGANIC POISONS IN BODIES.

By Dr. Anton Seyda.*

Introduction.

Although toxicologists have at their disposal a vast amount of chemical literature, I hope this article will be welcome to many, particularly as it is the result of my own experience and observations. My object has been to find a practical qualitative and quantitative course for the detection of those poisons which are within reach of the public, without troubling about such bodies as bitters, drastics, etc., which, in the present state of science, cannot be detected with certainty, whatever care and material the analyst might waste on the matter.

The analysis consists of a preliminary examination, and the regular chemical course. The expert conducting such an investigation must never omit to make the fullest inquiries about the case, as often valuable clues will be obtained, saving an immense deal of time and trouble.

Preliminary Tests.

(a) *Blood.*

The blood is often in a state of partial decomposition, particularly if a long time has elapsed between the time of death and the investigation. The reduction of its colouring-matter is more or less complete, so that a spectroscopical examination is, of course, useless. If the blood should be dry, it may be dissolved by water containing a trace of caustic soda. The spectrum of a partially reduced blood mostly shows a continued broad absorption band, which is composed of three lines, viz., two of oxyhæmoglobin, with the hæmoglobin one between. Sometimes the band is accompanied by another line in the red part of the spectrum, which is due to either hæmatin or methæmoglobin.

If this fourth line is observed, the blood must be tested as to its reaction. If alkaline, this points to methæmoglobin; if acid, to hæmatin. The first case deserves but little attention, as a rule, unless there has been poisoning with hydrogen sulphide (which is not likely to originate from the putrefaction process), but the second points to the administration of acids, nitroglycerin, potassium chlorate, or ferrocyanide or nitrobenzol. These bodies will then have to be searched for in the urine and other parts of the body.

In most cases, the only question is to decide whether the blood contains carbonic oxide. If we have to deal with a blood whose oxyhæmoglobin has been completely converted into the carbonyl compound, without the reverting process having set in, the detection of the carbonic oxide is easy, chemically as well as spectroscopically. If the blood, however, only shows the lines of the non-reduced or partially reduced hæmatin, the detection of the carbonic oxide is not successful, and, in fact, not likely to be so.

Not unfrequently, a blood will have to be examined for carbonic oxide, when in contains carbonylhæmoglobin, oxyhæmoglobin and hæmoglobin, with perhaps methæmoglobin or hæmatin.

In such a case, the presence of the carbonyl compound can only be proved by adding ammonium sulphide, and noticing whether the absorption band moves towards the red part of the spectrum. The chemical testing with soda ley, with or without calcium chloride, is not trustworthy.

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* *Chemiker Zeitung*, slightly abridged.

(*b*) *Urine.*

In testing this, it must be noticed this fluid undergoes remarkable changes whilst the poison is acting; also that many poisons only then pass into it when their physiological action is over. Therefore ascertain, if possible, the quantity of the urine, then look at its colour, notice the smell, test for blood, albumen, sugar, also its behaviour towards barium chloride, both before and after addition of hydrochloric acid.

The urine may also contain volatile bodies, balsams, alkaloids, and poisonous metals; whilst if poisonous doses of potassium chlorate, iodide or bromide have been given they will be readily detected in this fluid.

(*c*) *The contents of the Stomach.*

After noticing the odour and reaction, try whether there is a phosphorescence in the dark. If the stomach has an alkaline reaction, any phosphorescence cannot be due to phosphorus, but is caused by a fungoid growth, then phosphorus only lights when the contents are acid. The reaction of the contents will, according to circumstances, be acid, alkaline, or neutral. Poisoning caused by corrosive .acids, caustic alkalies, or haloids, is sure to have been recognised by the medical man who conducted the post-mortem examination.

The odour will not give much information if the contents are much putrefied. But when they are apparently in a good state of preservation, I have often noticed a loathsome, sweetish smell in the case of a decided form of arsenical poisoning. If the contents are solid, it is best to take an aliquot part and digest it in a small beaker with spirits of wine, and finally wash the insoluble matter with ether. The residue must now be rigorously examined with the microscope, which will often show remnants of medicines, pills, or powders. The alcoholic solution may be tested for oxalic acid. In many cases the stomach has been washed out for the purpose of the post-mortem. In this case the sediment of the wash-water must be carefully examined. Many indifferent objects will often be found, such as particles of carbon, fragments of coffee, potatoes, grain, greens or flesh, also crystalline bodies, such as triple phosphate, if the stomach has an alkaline reaction. If seeds are found they must be well washed with water, alcohol and ether, sorted, weighed and examined. If particles of arsenious acid are noticed, they may, after being freed from adhering matter, be gently dried and weighed, or dissolved in solution of potassium bicarbonate, and titrated with iodine. When the microscopical investigation is over, it is, in many cases, advisable to take a little of the contents and test for arsenic first, as this is the commonest form of poison. If not found, another portion may be exhausted with water, and the filtrate be examined for soluble metallic salts, more particularly the chlorate, nitrate, iodide, bromide, ferro or ferricyanide of potassium.

2.—SPECIAL PART.

If the preliminary testing has given no particular indication, I start the chemical analysis. I first of all submit the organs to a fractional distillation. The residue left in retort is then exhausted with alcohol to dissolve out any alkaloids, and then afterwards chlorinised for the purpose of testing for metallic poisons.

VOLATILE BODIES.

The contents of each jar are examined separately. A portion is cut up into small pieces and put into a suitable retort containing water. The first distillation is done by immersing the retort in boiling water for several hours. I then add to the contents a little tartaric acid and once more distil, this time by means of a current of steam. In this way we get two fractions. The first contains the readily volatile bodies, such as alcohol, aldehyde, acetone, chloroform, nitrobenzol, turpentine, camphor, amines, or their sulpho-derivatives. In the second, the remnants of the first, further fatty acids, carbolic acid, hydrocyanic acid, phosphorus, etc. If chloral hydrate or potassium cyanide * are suspected, the organs are moistened with respectively caustic potash or sodium bicarbonate and distilled from the water-bath, but should it be necessary to conduct a steam distillation from an alkaline fluid, I prefer to first make an acid extract with water containing some tartaric acid, which is then filtered and made alkaline before distilling.

If the acid distillate is ready, I properly notice its reaction, colour, odour, degree of turbidity, and particularly its bulk, so as to be able to make a quantitative estimation of any substance (such as alcohol) which the distillate may contain.

THE FIRST FRACTION.

(*a*) Test with acid silver nitrate; (*b*) with ammoniacal ditto (aldehyde); (*c*) with nitroprusside, potash, and acetic acid (aldehyde, acetone). Then test with alkaline permanganate (*d*) as follows:—10 c.c. of distillate are mixed with a few drops of potash ley and 1 c.c. of a saturated solution of permanganate and allowed to stand for twelve hours in a closed vessel. If oxidisable bodies are present, reduction soon sets in and the permanganate gets decolorised. The filtrate is then tested for aldehyde with ammoniacal silver. A large increase in the amount of aldehyde points to the presence of alcohol. As is well known, an alkaline permanganate acts differently in the cold than at the boiling heat. In the cold the following reaction takes place (in presence of organic matter):—

(1) $K_2Mn_2O_8 = Mn_2O_3 + K_2O + O_4$

(2) $K_2Mn_2O_8 + Mn_2O_3 = 2\,MnO_2 + Mn_2O_3 + K_2O + O_3$

On boiling: $K_2Mn_2O_8 = 2\,MnO_2 + K_2O + O_3$

It is highly probable, although not yet proved, that in the cold the liberated oxygen will first act on any organic matter before peroxidising the manganic oxide.

(*e*) With iodine and potash ley as follows:—In a test-tube I introduce a pinch of iodine, then 10 c.c. of the fluid and 3 drops of a 30 per cent. potash, then heat to 50° C., when, owing to the formation of iodoform, the liquid will get more or less turbid. If the iodine is used up, a little more of it must be added, and its excess finally be removed by cautious addition of potash ley. As iodoform does not always separate in well-defined crystals, the best thing is to agitate the solution with ether and allowing the latter to evaporate. If after a microscopical examination of the residue there is still a doubt, Lustgarten recommends the treatment of the crystals with resorcin and potash to get rosolic acid. (*f*) With resorcin and potash (chloroform). I have slightly modified Lustgarten's test. In a tube introduce a pinch of resorcin, 10 c.c. of the distillate, 3 drops of a 30 per cent. potash ley, and warm gently, when the beautiful rosolic acid is soon formed. This test is more delicate than the isonitril test; then however powerful and characteristic the smell may be, it is often masked by the presence of amines. In regard to the prussian blue test (treatment of chloroform with alcoholic ammonia and potash), although when performed under pressure a good result may be obtained, this test is far inferior to the rosolic acid test. (*g*) Zinc dust and hydrochloric acid (nitro-benzol). 10 c.c. of distillate are mixed with an equal volume of 90 per cent. alcohol, a little zinc dust is added, then 1 c.c. of hydrochloric acid, and a drop of platinic chloride. After standing for three hours, the liquid is decanted from the undissolved zinc and the greater part of the spirit driven off on the water-bath. The residue is now diluted with water, rendered alkaline with potash, and shaken out with ether. The ether is evaporated and the residue tested for aniline with freshly prepared solution of chloride of lime, which will produce a blue colour should nitro-benzol have been present. (*h*) With hydrochloric acid and alcoholic solution of phloroglucin (ethereal oils). This test, proposed by Ihl for the detection of bodies of an aldehydic nature, will often give unreliable results when applied to the distillate of portions of bodies, but it is best applied to the distillate of the urine. Hager's test for oil of turpentine is best carried out as follows:—A pinch of guaiacum is dissolved in 1 c.c. of absolute alcohol and 5 drops of oil of lemon and the mixture boiled; 10 c.c. of distillate are added and the whole boiled once more. The test only succeeds when notable quantities of turpentine are present and the reagent must be quite fresh. If, however, the distillate should have no action on alkaline permanganate or solution of chromic acid, ethereal oils are out of the question.

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* Supposing there is also a ferrocyanide.

A quantitative estimation of these volatile bodies will not often be practicable, not so much because no proper processes exist, but chiefly on account of the scarcity of material. An exception, however, is the estimation of the alcohol. Any one who has often had occasion to quantitatively determine alcohol in poisoning cases will often wonder how it is so comparative little attention has been paid to this subject in the works on toxicology and even chemistry. Rarely will the contents of the stomach be in such a fresh condition and the amount of alcohol so large as to admit of the estimation of the alcohol by the usual process, viz., distillation and specific gravity, more particularly as the gravity may be altered by the presence of amines. Even addition of mineral acids or even platinic chloride and redistilling will not altogether remove the organic bases. The only way is to oxidise the alcohol to acetic acid and to estimate this volumetrically. I carry out this estimation as follows:—The distillate is once more distilled off. What passes over in the first quarter of an hour is collected into a receiver containing some dry potassium carbonate. The process is repeated until we finally have a distillate measuring about 10—20 c.c., mostly strongly ammoniacal. It is put into a high-pressure flask, mixed with 3 c.c. of a twenty per cent.

olution of potassium bichromate, and, after being placed in a freezing mixture, mixed ith about 10 c.c. of strong sulphuric acid, or more if the iodoform test has been very uccessful. After closing the flask it is heated in boiling water for eight hours. fter cooling, the contents are put into a flask (kept cool), supersaturated with otash, and boiled until every trace of compound ammonias has completely disppeared, which is readily ascertained by exposing to the vapours a piece of red tmus paper.

After acidifying the solution with sulphuric or phosphoric acid, it is distilled (by eans of introducing steam) until the distillate is no longer acid. The distillate, often easuring a litre, is titrated with $\frac{n}{10}$ soda, using *litmus* as indicator. The alcohol is then lculated from the amount of acid found, and it must then be re-calculated to the iginal volume of the distillate. To make sure the distillate really contained acetic id, the neutralised fluid is boiled down to a small bulk. An aliquot part is tested ith barium chloride, and should there be any precipitate, it must be collected and lowed for. Another portion is mixed with a few drops of alcohol and then with lphuric acid in excess. If after twenty-four hours there is a strong odour of acetic her, the presence of acetic acid, therefore the presence of alcohol is proved beyond oubt. Or we may apply Carstanger's well-known cacodyl test.

The principles of the method just described will be easily understood. I must, owever, point out the absolute necessity of thoroughly boiling the oxidised mixture ith potash, otherwise the process is bound to fail. The amines always pass into the lcoholic distillate, even if distilled from an acid solution. They are not acted upon by the xidising mixture, and, therefore, unless removed by boiling with caustic potash, they ould again get into the acetic distillate.

As already stated, not much importance seems to be attached to the qualitative and uantitative toxicological determination of alcohol; not so much because there were no eliable processes, but principally because it is only reckoned a poison in its pure state. hold, however, a quite different opinion. Think how alcohol in its various forms, from randy to methylated spirits, is within almost everybody's reach, and how easily it may e abused; not so much in the case of grown-up people, but helpless infants. Is it not well-known fact, that gin, for instance, is largely administered to infants to make them leep, and how many have not in this way been killed by their unnatural mothers, ithout these in the least being suspected? Are not two tablespoonfuls of corn brandy ften a *fatal* dose for a child of six months old? Is it not probable that among the wer classes this comparative safe way of infant poisoning is largely practised? The hemical detection of alcohol is beset with difficulties; then first of all it rapidly passes ut of the system, and it has also been proved that traces are actually formed in the iving organism, whilst it is even a product of putrefaction. I therefore must insist pon the quantitative estimation of the spirit, which, if the amount should be excessive, ill point to the use of alcoholic liquors, or even tinctures, or may be in other espects a valuable clue.

(*To be continued.*)

ON A VOLUMETRIC METHOD OF GENERAL APPLICABILITY FOR THE DETERMINATION OF COMBINED SULPHURIC ACID.*

BY LAUNCELOT W. ANDREWS.

OF the volumetric methods at present in use for the determination of sulphuric acid, none can be considered entirely satisfactory, although some of them are very useful for technical purposes. Several of these methods involve a double titration, some require three standard solutions, and others are only applicable in the absence of bases precipitable by sodium carbonate.

The author believes, therefore, that the presentation of a new method, applicable in the presence of magnesium, calcium, aluminum, zinc, manganese, iron (ferric), nickel, cobalt, and silver, which requires but one standard solution with a single titration, and which in precision is at least equal to the ordinary gravimetric determination as barium sulphate, while demanding much less time for its execution, will not be thought superfluous.

The process depends upon the following series of reactions: First, to the solution of a sulphate is added an excess of a solution of barium chromate in hydrochloric acid; second, the solution is neutralised with ammonia or calcium carbonate, and filtered; third, the filtrate is acidified with hydrochloric acid, potassium iodide added, and the free iodine titrate with decinormal sodium thiosulphate solution (1 c.c. = 12·654 mg. iodine = 2·662 mg. SO_3).

The barium chromate employed may contain barium sulphate, but must be completely freed from soluble chromates and from barium carbonate, nitrate or chloride, by prolonged washing, first with boiling water slightly acidified with acetic acid, finally with hot distilled water, until the washings give no precipitate with sulphuric acid, and (in a quantity of 100 c.c.) a barely perceptible reaction with hydrochloric acid, potassium iodide and starch paste.†

A suitable solution of the barium chromate is prepared by digesting it with hydrochloric acid containing 36 grms. acid in the litre. This solution will contain from 2 to 4 per cent. of barium chromate, according to the temperature at which it was prepared.

The reagent being ready, the analysis is performed as follows:—

The sulphate to be determined is diluted, if necessary, until it does not contain more than 2 per cent, at most, of sulphuric anhydride, is made approximately neutral, and is heated to boiling. While boiling hot, an excess of the barium chromate solution is gradually introduced, and the boiling continued one minute, or longer, if carbonates were present.

The precipitate of barium sulphate is always yellow from the barium chromate which is carried down with it, provided sufficient excess of the latter reagent was added.

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* *American Chemical Journal.*

† When barium chloride is precipitated with an excess of potassium chromate, the water with which the precipitate is washed will, for a long time, show a yellow colour. This is not, as has been supposed, due to the solubility of $BaCrO_4$, but to the fact that it obstinately retains soluble chromate. According to the author's observation, barium chromate is not sufficiently soluble in water to impart any colour to the latter. According to the mean of four concordant determinations, one million parts of water dissolve 15 parts barium chromate at 18° C.

Calcium carbonate, which must be entirely free from barium or strontium carbonates or calcium sulphate, is then thrown in small portions into the still hot liquid until no further evolution of carbonic anhydride is observed, and the boiling continued one or two minutes. The solution is filtered while hot, and the precipitate is washed with a small quantity of hot water until the washings are colourless, and a little longer. Under these conditions, the barium chromate has no tendency to run through the filter or to retain any soluble chromate. 75 c.c. of water will usually be found more than sufficient to effect the washing.

If, on the other hand, the solution is allowed to stand over night with the precipitate in it, calcium chromate will be retained and a longer washing becomes necessary. In this case the results may come somewhat too high, in consequence of the necessarily large amount of wash-water dissolving an appreciable quantity of barium chromate. Under normal conditions the error from this source is quite insignificant.

The filtrate, after cooling, is treated with a sufficient amount of crystals of potassium iodide (free from iodate) and with 5 to 7 c.c. fuming hydrochloric acid for each 100 c.c. of liquid. This amount of acid is enough to induce a prompt and complete reduction of the chromate, but not enough to decompose the starch paste. It is advisable to run in the decinormal sodium thiosulphate from a burette until the brown colour of the iodine is nearly discharged before adding the starch, and then to continue the titration slowly, with constant stirring, until the turning point is reached.

The above process must be modified in the presence of ferric, nickel, or zinc salts by using ammonia instead of calcium carbonate to neutralise the acid liquid. The solution in this case is to be made distinctly alkaline, and the excess of ammonia boiled nearly away before filtering. The necessity for this modification arises from the fact that if a ferric or nickel or zinc salt is boiled with a chromate and calcium carbonate, basic chromates of iron and nickel are precipitated, from which the chromic acid cannot be removed, or only with difficulty, by washing. Barium chromate, when precipitated from its acid solution by ammonia, does not possess the agreeable properties which it shows when thrown down by calcium carbonate. It is much more finally divided than in the latter case, tends to run through the filter, which must therefore be double or of very dense paper, and requires a longer washing to remove the soluble chromates which it more tenaciously retains; consequently, ammonia is only to be used when the employment of calcium carbonate is inadmissible. It is not practicable to precipitate the excess of barium chromate with sodium acetate from hydrochloric acid solution. Numerous experiments, the details of which it is not worth while to give, show that barium chromate is so soluble in hot dilute acetic acid that the results fall 3 to 5 per cent too high, while if precipitated cold and allowed to stand, the results are too low.*

The following analyses show the applicability of the method :—

I. Taken 10 c.c. of a solution containing 20·8984 grams. pure and dry ammonium sulphate per litre. Diluted to 50 c.c., treated with 15 c.c. $BaCrO_4$ solution with $CaCO_3$, as described above.

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*The method cannot be used in the presence of phosphoric acid, and, of course, all reducing agents, as well as bismuth and copper salts, must be excluded.

Required 47·60 c.c. $\frac{1}{10}$ $Na_2S_2O_3$ sol.$=$·1267 gram. SO_3 found. Calculated, ·1266 gram. SO_3; 100·08 per cent. found.

II. Taken 10 c.c. of the same ammonium sulphate solution and treated as in No. I. Required 47·60 c.c. $\frac{1}{10}$ $Na_2S_2O_3$ sol.

The volume of the solution before titration was 300 c.c. 100·08 per cent. found.

III. 20 c.c. of the same ammonium sulphate solution, treated as before, required 95·50 c.c. $\frac{1}{10}$ $Na_2S_2O_3$ sol.

Total volume of the solution, 400 c.c.; 100·37 per cent.found.

IV. Taken ·2980 gram. $CuSO_4 + 5H_2O$, small crystals obtained by triple recrystallisation of a commercial sample. The copper was precipitated from boiling sol. with $(NH_4)_2S$ (free from sulphate), solution boiled with acetic acid to expel H_2S, filtered and filtrate treated as above.

35·95 c.c. ^{1} $Na_2S_2O_3$ sol. required $=$ ·0957 gram. SO_3 $=$ 32·11 per cent. found. Theoretical per cent. $=$ 32·09.

V. Taken 10 c.c. $(NH_4)_2SO_4$ solution (see 1) containing ·20898 gram., diluted to 100 c.c., treated with 20 c.c. $BaCrO_4$ sol. as above and neutralised with ammonia. Required 47·30 cc. $\frac{1}{10}$ $Na_2S_2O_3$ sol. $=$ ·1260 gram. SO_3 instead of ·1266 gram. Found 99·52 per cent.

VI. Taken ·6485 gram. $(NH_4)_2Fe_2(SO_4)_4 + 24H_2O$. Treated as No. V. Required 80·65 c.c. $\frac{1}{10}$ Na_2S_2O sol. $=$ ·2147 gram. SO_3. Calculated ·2153 gram. SO_3. Found 33·11 per cent. SO_3. Theory 33·20 per cent.

VII. *a*. Taken 100 c.c. of the Iowa City water supply. Treated as No. V. Required 2·10 c.c. $\frac{1}{10}$ $Na_2S_2O_3$. sol.

b. Taken 200 c.c. ditto, ditto. Required 4·30 cc. $\frac{1}{10}$ $Na_2S_2O_3$ sol.

Found 56 mg. and 57 mg. SO_3 per litre. A gravimetric analysis gave 58 mg. SO_3 per litre.

It seems probable that an indirect determination of sodium and potassium would be possible by weighing these elements as sulphates and determining the sulphuric anhydride volumetrically as described. Experiments to test this are now in progress. If feasible, it is evident that it would often be more convenient than the usual indirect determination as chlorides, while free from some of the well-known sources of error involved in the gravimetric determination of sulphuric acid in the presence of alkalies.

It is both a duty and a pleasure for me to take this opportunity to express publicly my thanks to Mr. F. W. Spanutius, instructor in chemistry here, for the zeal and skill with which he has assisted me in the experimental part of this investigation. Some of the test analyses given above were performed by him.

ELECTROLYTIC SEPARATIONS.

BY EDGAR F. SMITH AND LEE K. FRANKEL.

(Read at the December Meeting of the Chemical Section of the Franklin Institute.)

THE study of the electrolysis of the double cyanides of cadmium, copper and zinc, enabled us to formulate conditions, by which the separations of cadmium from zinc (*American Chemical Journal*, 11, 352) and cadmium from copper (*Journal of Analytical Chemistry*, 3, 385) were possible, and in every particular satisfactory.

The ease with which cadmium was separated from zinc, and the very close results obtained with these metals, led us to apply the method to the separation of cadmium from cobalt and from nickel. Operating first with cadmium alone, we dissolved sufficient pure sulphate in water, so that 10 c.c. of the solution would contain 0·1688 grams. metallic cadmium. To this volume (10 c.c.) were added four and one-half grams. pure potassium cyanide, and the solution made up to 200 c.c. with water. A current yielding 0·4 c.c. O-H gas per minute was allowed to act upon the same for a period of fourteen hours. The deposited metal weighed 0·1686 gram., a difference of − 0·11 per cent. from the theoretical. A second trial, with conditions precisely analogous to those just mentioned, gave 0·1690 gram. cadmium, a difference of + 0·11 per cent. from the theoretical.

CADMIUM FROM COBALT.

In the third experiment the conditions were the same as before, with this difference, that an equal amount of cobalt was also present in the solution. The result of the electrolysis was 0·1689 gram. cadmium, a difference of + 0·05 per cent. from the required.

The fourth experiment, similar in every way to the third, yielded 0·1689 gram. cadmium, a difference of + 0·05 per cent. from the theoretical.

The cadmium was fully deposited on both occasions, and contained no cobalt.

CADMIUM FROM NICKEL.

Passing to the separation of cadmium from nickel, the results were so surprising that we give the same in detail, although negative in character.

	Cadmium present, in grams.	Nickel present.	Potassium Cyanide, in grams.	Total Dilution.	Strength of current in c.c. O-H gas per minute.	Cadmium found.
1	0·1688	100 per ct.	4½	200 c.c.	0·3 c.c.	0·1717
2	”	”	”	”	”	0·1702
3	”	”	”	”	”	0·1725
4	”	”	”	”	”	0·1750
5	”	”	”	”	”	0·1721
6	”	”	”	”	”	0·1737
7	0·1828	—	”	”	0·5 c.c.	0·1827
8	”	75 per ct.	”	”	”	0·1923
9	”	50 per ct.	”	”	0·15 c.c.	0·1841
10	”	”	”	”	0·5 c.c.	0·1882
11	”	25 per ct.	”	”	0·15 c.c.	0·1854

The period of time during which the current acted in each of the above experiments was sixteen hours. Nickel was always found in the cadmium deposit, while in many cases the precipitation of the cadmium was incomplete. The conditions were varied, yet the results were wholly unsatisfactory. By greatly increasing the quantity of cyanide, we discovered that the cadmium precipitation was retarded. Nickel when alone, and when under the conditions given above, would not deposit with the strength of current used by us. This behaviour is only another indication that if we would make electrolytic methods widely applicable, it is first necessary to extend the study of the action of the current to all the salts possible, and to investigate carefully the influence of each metal upon its associates under varying conditions.

From what we have thus far accomplished, we find the electrolytic separation of cadmium from copper, from zinc, and from cobalt, in cyanide solution, all that could be expected from any method. The cadmium deposits, in the experiments recorded in this paper, were always washed with boiling water; the drying was done upon the edge of a warm iron plate.

We have already called attention (*American Chemical Journal*, 11, 264) to the fact that mercury is fully precipitated from the solution of its double cyanide by a comparatively feeble current, and that the separation of this metal from copper is possible so long as the quantity of the latter does not exceed twenty per cent. of the mercury present.

More recently we have executed a series of experiments looking to the separation of mercury from zinc, nickel, and cobalt.

MERCURY FROM ZINC.

The results with these metals are :—

	Mercury present in grams.	Zinc present.	KCN in grams.	Total dilution.	Current strength in c.c. O-H gas per minute.	Mercury found.	Difference in per cent. from the theoretical.
1	0·1715	—	4½	200 c.c.	0·3 c.c.	0·1717	+0·12 per cent.
2	,,	—	,,	,,	,,	0·1715	—
3	,,	100 per ct.	,,	,,	0·25 c.c.	0·1706	- 0·52 per cent.
4	,,	,,	,,	,,	,,	0·1709	- 0·34 per cent.

The time in each deposition was sixteen hours. From these figures the separation is possible. Mercury was not found with the zinc. In the following experiments it will be observed that the error is much less, and accordingly makes the method trustworthy and, from its accuracy, well suited for scientific as well as technical work :—

	Mercury present in grams.	Zinc present.	KCN in grams.	Total dilution.	Current strength in c.c. O-H gas per minute.	Mercury found.	Difference in per cent. from the theoretical.
5	0·2440	100 per ct.	3	200 c.c.	0·5 c.c.	0·2435	−0·20 per cent.
6	,,	,,	3	,,	,,	0·2445	+0·20 ,,
7	,,	,,	4½	,,	,,	0·2441	+0·04 ,,
8	,,	,,	,,	,,	0·28 c.c.	0·2445	+0·20 ,,
9	,,	,,	,,	,,	,,	0·2431	+0·37 ,,
10	,,	,,	,,	,,	,,	0·2454	+0·50 ,,

The time of precipitation, made at the ordinary temperature, amounted to sixteen hours. The mercury deposit was washed with hot water, and dried upon a moderately warm iron plate. (*To be continued.*)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Estimation of Uric Acid by Sodium Hypobromite. M. Bayrac (*Compt. rend.*, 7 Feb., 1890).—50 c.c. of the urine are evaporated on the water-bath. The residue s treated with 5 to 10 c.c. of diluted hydrochloric acid (1 in 5), and the precipitate is ashed with alcohol to remove urea and creatinine. It is then dissolved on the water-ath with 20 drops of solution of sodium hydrate. and heated to nearly boiling with 5 c.c. of saturated solution of sodium hypobromite in the usual apparatus for urea stimations. The whole process can be performed under two hours. M. S. A. M.

The Ferric Chloride Test for Cocaine. Prof. Plugge (*Ned. Tydschr. v. harmacie, etc.*, March, 1890).—Messrs. Lerch and Schärges have lately published a elicate test for this alkaloid. A drop of very weak ferric chloride is added, and the ixture heated to boiling. A blood-red colour is developed, almost resembling ferric lphocyanide. And apparently nothing is more natural. Does not cocaine on boiling ith water decompose into ecgonine, methylic alcohol, and *benzoic acid?* And does ot this acid give a red (?) colour with ferric chloride. How scientific this may all seem, he author found, however, that the same colour may be just as readily got by pplying the test *without cocaine*, for reasons which any one acquainted with the hemistry of iron salts will readily understand. L. de K.

New Test for Conine. Van Senus (*Ned. Tydschr. v. Pharmacie, etc.*, March, 890).—This alkaloid gives a fine blue, changing to red and yellow when mixed with itro-benzol. Aniline and nicotine do not give the reaction. The test is, however, not uccessful with the pure $C_6H_5NO_2$, but only with the article prepared by acting with itric acid on *commercial* benzol. When such nitro-benzol is distilled, the first fractions ive the reactions but very imperfectly, but the residue left in the retort produces a ine display of colours. The author soon hopes to be able to find out to what substance he reaction is really due. Not unlikely it may be caused by the presence of carbonic ulphide in the crude benzol. L. de K.

Adulteration of Linseed Oil. Prof. Wefers Bettink (*Ned. Tydschr. v. harmacie, etc.*, March, 1890).—A sample submitted to the author, although conforming o the standards of purity as at present laid down, was, however, perfectly useless for ainting purposes, as when mixed with white lead this became brittle in a few ours. This pointed to a large amount of free acid, which induced the author to pply the process of Salskowski, viz., dissolving in ether-alcohol and titurating with $\frac{n}{0}$ alcoholic potash. Thirty-four per cent. of free (oleic) acid was thus found. To scertain the nature of the acid, a large quantity of the oil was agitated with a 10 per ent. solution of common soda. After the greater part of the oil had separated it was gitated with petroleum spirit to free it from oily matter, and then decomposed with cid. The fatty acids thus separated gave a I. absorption of 146. (No doubt this figure ould have been higher still if any stearic acid had been first removed.) The author hesi- tes to call it linoleic acid, but feels pretty sure it has been wilfully added. L. de K.

Estimation of Free Acid in Solutions of Stannous Chloride. W. Minor (*Zeitschr. f. angew Chemie*, No. 1).—The estimation of hydrochloric acid in solutions of stannous chloride cannot be done with silver nitrate, as insoluble stannic hydrate would be formed as well as silver chloride. The author recommends the following process:—

10 c.c. of the fluid are diluted with hot water, and hydrogen sulphide is passed until all the tin is precipitated. The liquid is filtered off from the precipitated stannous sulphide in a litre flask. After making up the contents to the mark, 500 c.c. (= 5 c.c. original fluid) are boiled for a short time to expel the hydrogen sulphide, and finally titrated with normal soda. This will give both free and combined hydrochloric acid.

The amount of combined acid may of course be readily calculated from the amount of stannous tin, which the author prefers to estimate with iodine. If the sp. gr. of the fluid is known, the amount of acid by weight can be readily calculated.

L. de K.

Artificial Coffee in Germany. R. Wolffenstein (*Zeitschr. f. angew Chemie*, No. 3, 1890).—The author analysed two varieties, known in Germany as Domkaffee and Allerweltskaffee. The complete absence of caffeine undoubtedly proved the absence of coffee. The microscope showed the first article to practically consist of chicory, whilst the other one contained large quantities of lupines. The author also succeeded in isolating a brown colouring matter, possessed of the same properties as the well-known Casseler-brown. It not only behaved spectroscopically the same, but also in its chemical reactions. Although soluble in alkalies and in water, it is completely precipitated by addition of mineral acids, such as hydrochloric acid. 14 grms. of the sample were extracted with water and precipitated with hydrochloric acid, which yielded 1·67 grm. of colouring matter, which corresponds with 11·9 per cent. adulteration.

REVIEWS.

Chemical Laboratory Labels; compiled by W. H. Symons, F.C.S., F.I.C. Second Edition, Part III.

This is a collection of labels for large bottles, supplementary to Parts I. and II. already published, which latter are for smaller bottles. The labels are well printed, well selected, and really cheap, there being 250 in this book for 1s. 6d. Tables for conversion of thermometric and barometric degrees, and for the pressure of aqueous vapour are also included, ready for sticking up in any convenient place. All Mr. Symons' volumetric labels are very well done, with a full list of equivalents on each, and his collections are certainly the best in the market at the moment for the practising analyst.

Catalogue of Scientific Apparatus and Chemicals; by J. Orme and Co., 65, Barbican, E.C.

Messrs. Orme have just issued a very complete and well-illustrated catalogue of chemical and physical apparatus and chemicals, a special feature of which is the consideration of all articles for photography. The prices are reasonable, and the book should add to the business done by the firm, as well as being convenient for reference by its possessors.

THE ANALYST.

MAY, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON THE DETERMINATION OF RESIN IN SOAP.

BY ROWLAND WILLIAMS F.I.C., F.C.S.

Read at Meeting, March, 1890.

SEVERAL chemists have devised methods of greater or less value for the purpose of estimating resin in soap and in fatty substances generally, but few of these can be regarded as really satisfactory. Among the processes in vogue at different times may be mentioned those of Barfoed and Sutherland. The former of these depends upon the alleged practical insolubility of dry soda soaps (made from ordinary fatty substances) in a mixture containing five parts ether and one part alcohol, while resinate of soda is said to dissolve easily in this solvent.

Sutherland's method consists in oxidation of the resin by means of nitric acid, which is assumed to remove resin without attacking fatty acids. I have not found either of the above methods at all reliable, more especially the latter. Indeed, until a few years ago, it can hardly be denied that it was impossible to separate resin from complex mixtures containing various kinds of fatty matters, with even an approximate degree of accuracy.

In 1882, however, Mr. T. S. Gladding introduced his excellent method for the determination of resin in various fatty substances, and it is with this method that my present remarks are chiefly concerned. Mr. Gladding described his method originally in the *American Chemical Journal* (Vol. III., No. 6.), and a full description will also be found in "Allen's Organic Analysis" (Vol. II., page 78, et seq). The process in question is probably so well-known to most of you that it would be presumption on my part to dwell at length upon the modus operandi. I may say, however, for the benefit of any who may not be very familiar with the subject, that the method is based upon the fact that silver resinate is soluble in ether containing a certain proportion of alcohol, while silver oleate, stearate, etc., are practically insoluble in such a menstruum. Three or four years ago, Alder-Wright and Thompson (*Chemical News*, Vol. 53, page 165), submitted Gladding's process to a careful investigation, with the result that, on the whole, they are satisfied with its essential accuracy. The main point upon which they differ from Gladding is with regard to the solubility of certain fatty silver salts, which they find in some cases to be greater, and in others less, than stated by Gladding in his original paper.

I have recently had a good deal of experience of Gladding's process as applied to the determination of resin in soap. and have found it very satisfactory. I find it best to work on the soap itself, instead of on the fatty acids separated therefrom, and have no difficulty in obtaining good results by slightly modifying the original process in one or two details, which will readily suggest themselves to anyone with a little practice. As I believe there is no record of experiments made with Gladding's process on soap containing known proportions of resin, the following results are interesting as confirming the accuracy of the method.

Some time since I received for analysis a sample of soap, in which an estimation of the resin was required. The firm who sent the soap knew approximately the percentage of resin present, but I was not aware until afterwards that this was a test case. I reported that the soap contained 30·7 per cent. of resin. One of the parties concerned subsequently informed me that he had calculated it to contain 30 per cent., so my estimation was considered very satisfactory. Since then, two of the largest soapmaking firms in this district have been so kind as to supply me with samples of soap containing moderate amounts of resin, at the same time informing me of the proportions of fatty matters and resin employed in the manufacture thereof. I was told that one of these samples, which I will term "A," had been made from a mixture of 100 parts tallow and twenty-five parts resin by weight, and the other, "B," from a mixture of seven parts fats (nature not specified) and two parts resin. Calculated from the percentages of mixed fatty and resin acids respectively present in the two soaps, the theoretical amounts of resin would be 12·84 per cent. in "A," and 13·82 per cent. in "B."

On examining the two samples by Gladding's method I found the following percentages of resin:—

	Found.	Calculated.
"A"	13·17 per cent. ..	12·84 per cent.
"B"	14·06 " ..	13·82 "

These results prove the reliability of Gladding's method, at any rate in the case of ordinary hard soaps.

It may be interesting to mention that I recently heard of a certain chemist (a well-known member of our Society) stating as the result of his analysis of a much advertised cheap soap, that it contained only about one-half the amount of resin which his clients knew to be present. This circumstance alone is, I think, sufficient to show that a little more light is desirable on methods employed for determinations of resin. If my paper to-night should prove the means of attracting more attention to this *important matter*, I shall feel amply repaid for bringing the subject before you.

N THE SOLUBILITY OF PHOSPHATE OF ALUMINA IN ACETIC ACID WITH SPECIAL REFERENCE TO THE ESTIMATION OF ALUMINA IN FLOUR, BREAD, ETC.

By W. C. Young, F.I.C., F.C.S.

(*Concluded from page* 63.)

·1 per cent. alum solution used:

	A.	B.	C.	Mean.	
1.	·0215	·0235	·0235	·0228	gramme.
2.	·0215	·0235	·0240	·0230	,,
3.	No ppt.	No ppt.	No ppt.		
4.	·0165	·0145	·0190	·0167	,,
5.	·0205	·0150	·0200	·0185	,,

·2 per cent. alum solution used:

	A.	B.	C.	Mean.	
1.	·0445	·0450	·0445	·0445	gramme.
2.	·0460	·0465	·0465	·0463	,,
3.	No ppt.	No ppt.	No ppt.		
4.	·0235	·0225	·0230	·0233	,,
5.	·0400	·0400	·0410	·0403	,,

ese results show (except in experiment No. 1 with the ·1 per cent. solution of alum, here the quantity obtained was practically the same as when 5 c.c. of acetic acid were ed) that the increase of acetic acid had a decided effect on the solubility of the phos- ate of alumina, especially when precipitated in the cold, or when, after precipitation boiling, it remained in contact with it in the cold for some hours.

In determining the quantity of alum present in adulterated bread or flour by upré's process or its modifications, there is present during the process either chloride ammonium or sodium in addition to the salts used in these experiments, and as the rmer might possibly influence the precipitation of phosphate of alumina, I repeated the ries of experiments, adding in each 10 c.c. of a 20 per cent. solution of chloride of nmonium, the result obtained being as follows:—

·1 per cent. solution of alum as before, with 5 c.c. acetic acid and 10 c.c. of a 20 er cent. solution of chloride of ammonium:

	A.	B.	C.	Mean.	
1.	·0245	·0230	·0230	·0235	gramme.
2.	·0250	·0250	·0250	·0250	,,
3.	No ppt.	No ppt.	No ppt.		
4.	·0215	·0215	·0225	·0218	,,
5.	·0245	·0225	·0235	·0235	,,

·2 per cent. solution of alum as above:

	A.	B.	C.	Mean.	
1.	·0475	·0485	·0485	·0482	gramme.
2.	·0475	·0485	·0475	·0478	,,
3.	No. ppt.	No ppt.	No. ppt.		
4.	·0425	·0440	·0430	·0432	,,
5.	·0450	·0465	·0455	·0457	,,

·1 per cent. solution of alum with 15 c.c. acetic acid, the rest as above:

	A.	B.	C.	Mean.	
1.	·0220	·0225	·0225	·0223	gramme.
2.	·0245	·0245	·0235	·0242	„
3.	No ppt.	No ppt.	No. ppt.		
4.	No ppt.	No ppt.	No. ppt.		
5.	·0155	Turbidity	·0195	·0175	„

·2 per cent. alum solution, the rest as in last series:

	A.	B.	C.	Mean.	
1.	·0405	·0415	.0405	·0408	gramme.
2.	·0425	·0415	·0445	·0428	„
3.	No. ppt.	No. ppt.	No. ppt.		
4.	No. ppt.	No. ppt.	No. ppt.		
5.	·0365	·0345	·0360	·0357	„

From these results it will be seen that the addition of the chloride of ammonium to the experiments in which 5 c.c. of acetic acid were taken completely prevented the immediate precipitation in the cold in every case, lessened the amount of precipitate obtained after standing some hours, and increased the quantity dissolved by standing in the cold after precipitation by boiling, but had little effect upon the precipitation when collected immediately after boiling. In the experiments in which 15 c.c. of acetic acid were taken, the addition of chloride of ammonium completely prevented the precipitation in the cold, even after standing over-night, and materially lessened the precipitation in every other case.

My object in arranging the experiments in the manner described was to produce, as nearly as I could, the conditions existing in Dupré's method and the various modifications since suggested.

Dupré insists upon precipitation in the cold, using a small excess of acetic acid, Wanklyn precipitates boiling with a large excess of acetic acid, separating the precipitate immediately, and Bell boils and sets aside for several hours, using a small excess of acid.

The results of my experiments show that the nearest approach to the truth is obtained when the precipitate is separated immediately after boiling; but it is generally admitted that by so doing some phosphate of calcium is obtained with the phosphate of alumina.

I have a strong opinion that the temperature has some influence upon the quantity of precipitate obtained in the cold, or when the precipitate has been allowed to stand in contact with acetic acid in the cold, after precipitation by boiling, and regret that I have not had time to put the matter to the test, but I have noticed that the quantity obtained in cold weather is less than in hot.

I may mention that rye bread, of which I frequently have samples for analysis from the east end of London, invariably gives a strong reaction with a logwood test, although I have never found more than ·008 gramme. phosphate of alumina in 100 grammes.

(*Conclusion of the Society's Proceedings.*)

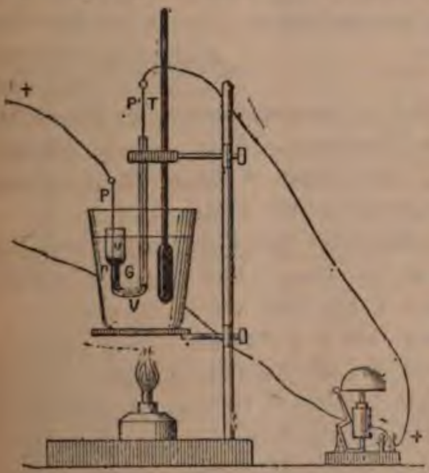
METHODS AND APPARATUS IN USE AT THE LABORATORY OF THE BOURSE DE COMMERCE AT PARIS FOR THE ANALYSIS OF CERTAIN COMMERCIAL ORGANIC PRODUCTS.

By Dr. Muter.

Some years ago we gave an account in the Analyst of the methods in use at the Paris Municipal Laboratory, and we think that many of the readers of the journal will be interested to know how things are managed at the new laboratory of the Bourse, under the direction of M. Ferdinand Jean. Many of the methods and apparatus in use at this establishment are the invention of the director, and have the merit of the greatest ingenuity. This is specially noticeable in the analysis of fats and of tanning materials, with both of which subjects we propose to deal in the present article.

(1) Apparatus for Taking the Melting-point of Fats.

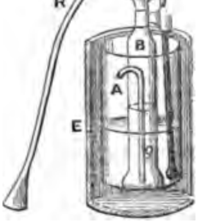
In this arrangement the melting-point of the fat is taken as being the degree at which it is sufficiently fluid to permit of the sinking down of mercury placed on the surface of the solid fat and the establishment thereby of an electric contact, which in turn rings a bell at the exact moment of contact. The drawing given herewith will easily explain its action. The essential part is the "U" tube G (having the form shown) which is held, together with the thermometer T, in a vessel of water over a source of heat. The fat, having been melted at the most gentle possible heat, is poured into the "U" tube by the shorter limb so as just to fill the bend. Before it has time to solidify two platinum wires are pushed down so that a wire reaches well into the bend on each side, but the two wires do not quite touch each other. Some mercury is now placed on the top of the solidified fat in the shorter limb, and the tube and thermometer are placed *in situ*. One of the platinum wires is then connected to one side of an electric bell, and the other to one pole of a galvanic cell, the other pole of the cell being connected to the other side of the bell to complete the circuit. Heat is then cautiously applied and, at the moment of fusion, the mercury sinks, establishes contact, and the bell rings. The degree marked on the thermometer at the instant the bell rings is the melting-point of the fat. The apparatus is certainly very sensitive, and it is possible to act on samples of the same fat again and again, and obtain results that are exactly concordant. Of course, with such an arrangement, one must do as has been done in the laboratory and establish one's own standard for the melting-points, as the results will not always agree with recorded figures by the ordinary methods of taking melting-points. M. Jean lays the greatest stress on the necessity of always avoiding the fusion of fats to more than a degree or two over their melting-point before submitting them to examination, as he is a strong believer in the doctrine that fusion



of fats (especially of those like butter) completely alters their proximate molecular arrangement. He argues that many of the discordant results obtained by various observers of the physical properties of fats (such as density, melting-point, etc.) are entirely due to such rash and prolonged fusion before analysis.

In the analysis of tallows, palm oils, etc., the solidifying point of the fatty acids separated after saponification is considered a strong point. This is done in test tubes about 12 centimetres long and 15 millimetres in diameter, which are half filled with the fused acids, and a delicate thermometer (graduated in tenths of a degree) is introduced so that it goes well down into the tube but does not touch the bottom. This tube is placed in an outer jacket to prevent too rapid cooling, and the whole is watched. The thermometer sinks regularly at first until crystals of fatty acid begin to form on the surface and on the sides of the tube, and then, for a moment or two, the thermometer ceases to sink. At this point the fat is slightly agitated by turning the thermometer three times to the right and then three times to the left, when the temperature will be noticed to rise several tenths of a degree and then again become stationary. It is this latter point that is considered to be the true solidifying-point of the fatty acids, and the standards of tallow, etc., are set thereon.

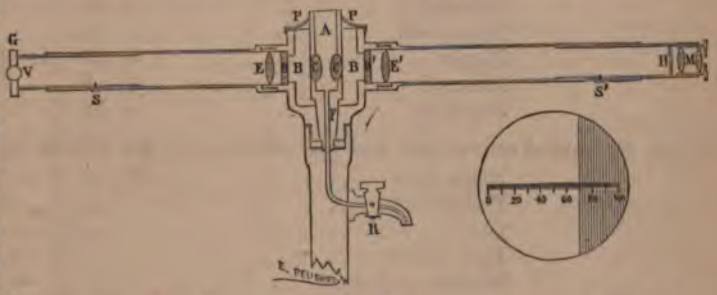
(2) APPARATUS FOR TAKING THE HEATING POWER OF OILS WITH SULPHURIC ACID.



The construction of this apparatus (called a thermelæometre by M. Jean) will be easily understood by reference to the illustration. A is a little glass cylinder having a graduation at the height of 15 c.c.; B is a specially constructed tube for containing the acid, having a perforated stopper connected to an india-rubber tube and mouthpiece. A little delivery tube is fused into its side, so that if the operator blows gently into the mouthpiece, Y, any fluid contained in the tube is ejected through the delivery tube at a regular rate of speed. T is a thermometer dipping into the oil cylinder, and E is a non-conducting outer casing to prevent the loss of heat The acid used is a commercial acid of 1·819 specific gravity, which is kept in a large bottle fitted with one of M. Jean's automatic burettes, by which the acid can be measured into the apparatus without any exposure to the air, and in which it may be stored for any length of time without absorbing any moisture or decreasing in strength. To use the apparatus, 5 c.c. of acid are introduced into the inner tube and the stopper securely fixed. Fifteen c.c. of the oil are then placed in the cylinder, and the acid tube having been grasped by the hand, the oil is stirred thereby (applying heat or cold, as the case may be) until the thermometer marks 20° Centigrade. The whole is then placed in the outer non-conducting case, and, by blowing into the mouthpiece the acid is caused to mix with the oil (the acid tube and thermometer being meanwhile used as a stirring arrangement) until the thermometer marks its maximum. In the case of drying oils, 5 c.c. of a retarding mineral oil of ·860 specific gravity are first added to the 15 c.c. of oil, and the effect of this addition is allowed for by the usual calculation. It is remarkable how handy this little piece of apparatus is, and what really concordant results can be obtained by its use time after time with different experiments on the same oil. If the oil to be examined be at all rancid, it is invariably shaken up several times with hot alcohol to remove free fatty acids and then run off from the separator in which the shaking was conducted and dried at 110° Centigrade. In actual practice all oils are really thus treated before taking their heating power, and where the oil is very stale indeed, it is saponified and the heating power of its fatty acids is taken, but in this case they begin working from the initial temperature of 30° Centigrade. In determining the nature of an oil or fat that has been already saponified (such as in a soap) the acids are liberated, dried at 110° Centigrade, and treated in the apparatus at the initial temperature of 30° Centigrade. There are, according to M. Jean, many oils showing very little difference on the original oil, but yet exhibiting marked differences when the process is applied to their fatty acids.

3. The Oleorefractometre and its Uses.

The analysis of oils and fats is practically carrried out by three observations: (1) the density; (2) the thermelæometre as above described; (3) by the important instrument with which we are now about to deal, the construction of which will be evident on studying the drawing.



The centre of the instrument is formed by a circular metallic receptacle, B B, closed by two opposite lenses, E E, from which extend two tubes, S S, the one ending in a collimator, G, and the other in a short-visioned telescope, M. In the middle of the receptacle is placed an inner metallic reservoir, A, having glass sides placed at a suitable angle, so as to form a prism. In front of the field-glass of the telescope is a photographic scale, H, and there is also a moveable vertical shutter, so placed as to divide the luminous field of the instrument into two parts, the one being quite bright, while the other is in shadow. It is the position of the edge of this shadow on the scale that determines the readings with the apparatus. If the same liquid be placed in both reservoirs the shadow will come on the zero of the scale; but if a different liquid be placed in the inner prismatic reservoir, the shadow will come at some point either to the right or to the left of the zero, according to the nature of the refractive power of the liquid. The appearance of the shadow in the field is shown in the drawing. The illumination is obtained by an ordinary gas jet placed opposite to the collimator end of the instrument. Both receptacles are furnished with draw-off cocks, and are enclosed in an outer case (not shown in the drawing), into which water is put for the purpose of regulating the temperature, and which case is heated by a little lamp placed underneath, so as to maintain its contents at any desired degree. M. Jean has invented a typical oil that gives no refraction, and with which the outer receptacle is always filled, and the temperature is brought to 22° C. If now some more of this oil be also brought to 22° C, and placed in the prismatic reservoir, the shadow will mark zero, or if it does not do so it is adjusted to that point by moving the shutter. The typical oil is then run out of the prism, and the oil to be tested, having been brought to 22° C., is placed therein and the reading is taken. All the vegetable oils, and some marine animal oils, deviate the shadow to the right of the zero, while the terrestrial animal oils act in the contrary sense. It takes about half an hour to regulate the instrument and get all the temperatures equal in the reservoirs, but once this is attained, one sample after another can be done at intervals of five minutes, a portion of every sample of oil being used to rinse out the inner prismatic reservoir before actually filling it for observation. The following table will give some idea of the differences that can be got between the various vegetable oils:—

Olive	+ 1·5 to + 2
Colza	+ 16·5 to + 17·5
Ground-nut	+ 4·5
Sesame	+ 17·0
Cotton	+ 20·0
Castor	+ 40
Linseed	+ 53
Hempseed	+ 33
Poppy	+ 30 to + 34
Almond	+ 6
Japonica	+ 50

Of the terrestrial animal oils and fats which go to the left we have:—

Neats-foot	— 3
Horse-foot	— 12
Lard	— 12·5
Beef tallow	— 16
Mutton tallow	— 20
Butter fat	— 35
Margarine (average)	— 15
Oleic acid	— 34

Before observing any oil, it is to be, if at all rancid, shaken up in a separator with hot alcohol, to remove the free fatty acids, and dried at 110° C. This is especially necessary in the examination of the lower qualities of olive oil, and indeed, with this particular oil, it is always desirable to proceed in this manner, if a preliminary examination of the oil itself does not indicate the proper refraction.

In applying the oleorefractometre to solid fats, such as butter and lard, it is necessary to work at 45° Centigrade. The butter is first melted at the lowest possible temperature and strained through muslin. It is then dissolved in ether, and the ethereal solution is shaken up in a separator with tepid water. The solution is then transferred to a flat-bottomed flask, the ether is distilled off, and the residual fat is slowly heated up to 110° C. till quite dry. Lastly, it is cooled down to 45° C and examined in the refractometre. Thus treated, butter gives a refraction of 35 to the left, while margarine gives an average of 15 in the same direction. M. Jean has met with exceptional butters from a single cow's milk in which the refraction has fallen to 33, but out of the immense number of samples examined annually in the laboratory, all the genuine butters come up to the 35 with very few exceptions, and at the Bourse laboratory they consider that they can always certify with confidence to any amount of adulteration over 10 per cent. We tried to take an observation on some samples of exceptional butter fat given us by Dr. Vieth last spring; but they were entirely decomposed and came out nearly tallow. On the question of decomposition, the custom over there is to entirely refuse to give an opinion on a sample of butter unless it is quite fresh. The use of vegetable fats in the manufacture of margarine renders its detection by the instrument exceedingly easy, because the least quantity of such fat sends the refraction down tremendously. American lard adulterated with vegetable oils is very readily caught by the apparatus, because of the great variation in the refraction of lard (— 12·5) and that of cotton oil (+ 20).

The use of the instrument is not, however, limited to the analysis of fats, but it is also employed for the detection of methylated spirit as follows:—Pure spirit, of exactly the same density as the sample to be examined, is put into the outer receptacle, and the inner one is also charged with the same spirit and the zero set. The standard spirit is run out of the prism and its place is filled with the sample, when a large left-hand deviation will be shown if methyl alcohol be present. Ten per cent. of methylation will show as much as 13 degrees. No mistake can be made between methylated and impure spirit, because the presence of fusel causes a refraction on the right.

The instrument is also employed in the analysis of oil of turpentine in a similar manner. It is first set with good oil of turpentine, and then the genuine is run out of the prism and the sample is substituted. The presence of either light oil of tar or of petroleum spirit, is at once shown by the powerful left-handed refraction produced. Thus, 10 per cent. of light oil of tar will produce a deviation of something like 40 degrees, while the same amount of petroleum spirit will deviate about 15.

M. Jean is now engaged on researches on the various essential oils employed in perfumery, and he has good hopes of soon placing chemists in a position to put a stop to the great amount of fraud that now exists in this trade, owing to the want of good processes and standards.

The whole working with this instument is at the moment in its infancy, but I fancy it will one of these days become an indispensable adjunct to every commercial laboratory. At the present time its cost (£16) is somewhat prohibitory. It is made by Duboscq.

Before leaving the subject it will be interesting to state that the instrument has been put to the test by a committee of the French Agricultural Society. They had six samples made up of pure butter from various sources, and also of butter adulterated with margarine. These were examined by M. Jean in the presence of the Committee, and he was found to be correct in every case within ten per cent. of the admixtures, while all the pure butters were correctly recognised and duly passed as such.

Whatever be the cause of the varying refractive powers of oils and fats, it evidently does not vary with their density, because we find oils whose densities are very similar and which yet exhibit totally different refractions. It is this point that makes the joint determination of the density and of the refraction so useful. Supplement this with a really accurate determination of the heating power and, when necessary, with the Reichert and iodine process, and it would almost seem as if we were at last on our way to the real power of distinguishing between the various oils and fats.

(*To be continued.*)

DETECTION AND QUANTITATIVE ESTIMATION OF ORGANIC AND INORGANIC POISONS IN BODIES.

BY DR. ANTON SEYDA.

(*Continued from page* 73.)

THE following observations, which I have made during my search for alcohol in the various organs from persons who were confirmed drunkards, will be of interest. The organs did, however, not reach me for a week or a fortnight. 1. Pure ethylic alcohol was found in a few cases. From the contents of each jar containing respectively the stomach, duodenum small intestine; the pieces of the large glands of the abdomen; the lungs and brain, I have always succeeded in getting alcoholic distillates, but I never succeeded in getting alcohol or aldehyde from the urine. In many cases (it was not known whether the persons had partaken of alcoholic liquors shortly before death) I noticed the interesting fact, that whilst the distillate from the stomach was absolutely free from alcohol, the liver, kidneys, spleen, yielded a distillate containing it. In one case the reaction of the stomach was acid, and in the other faintly alkaline. The distillates when containing alcohol, gives the following reactions:—

1. Alkaline permanganate is reduced in the cold; the filtrate reduces ammoniacal silver nitrate.

2. A mixture of potassium bichromate with sulphuric acid is also reduced. The mixture has either the odour of aldehyde, acetic acid, acetic ether, or a pungent odour cf rum, or often of a fruit essence.

3. A more or less strong iodoform reaction will be got.

4. If a few drops are collected at the beginning of the second distillation (over dry potassium carbonate), these will burn with the characteristic alcohol flame. This beautiful (but not very conclusive) test may even succeed when the organs are in a advanced state of putrefaction, but it will have to be conducted with great care, and best in a dark room. Before setting light to it, the crucible containing the spirit must be slightly warmed. The drawback to this test is the waste of the greater part of the spirit.

The cases where alcohol was only obtained from the liver and spleen, whilst none could be got from the stomach, etc., seem to me very peculiar. It is not improbable that the alcohol was formed by a kind of fermentation of the glycogen contained in the liver. It is also difficult to explain how it came that in one case where the medical men conducting the post-mortem, recognised alcohol by its smell, and certified as to alcoholic poisoning, no ethylic alcohol was found, but its higher homologues. This, however, was after a lapse of eight days between the inquest and the analysis. This strange fact may perhaps be explained by the great volatility of ethylic alcohol, causing it to evaporate even from the dead organism. The following experiments which I made to show the volatility of even dilute alcohol, proved interesting:—

In a beaker 75 m.m. square, I put some very dilute alcohol, and allowed it to spontaneously evaporate, estimating the percentage of alcohol from time to time by means of the vapourimeter. The results were as follows:—

On 20th December, 1886, the liquid contained 2·4 volumes of alcohol.
" 21st " " 2·0 "
" 24th " " 1·4 "
" 27th " " ·75 "
" 28th " " ·60 "
" 10th January, 1887, " ·20 "
" 22nd " " ·0 "

eaker still contained water.

rom these experiments it is plain that alcohol rapidly evaporates even from dilute ns. If one now considers how clumsily post-mortems are sometimes conducted, it arcely be a matter of surprise, the alcohol has in many instances completely ated before the organs reach the analyst. In conclusion, I may mention that the quantity of alcohol I ever found in a stomach did not exceed ·2 gram., whilst her organs yielded still less of that substance.

THE SECOND FRACTION.

(*a*) *Carbolic Acid.*

s this body is always among the products of the putrefaction of albumenoids, one nerally find a trace of it with Millon's reagent* or (though not so frequently) romine water. If a poisonous dose is present, its amount may be estimated in a ctory manner as follows :—

he distillation is carried on until what passes over, gives no colouration with ferric le. Not to lose any, it is advisable to continue the distillation until no colouration by Plugge's test. Both distillates are now filtered off from any fatty matter, and tely shaken out with *pure* ether. The joint ether is now allowed to evaporate, e residue is titrated in the well-known manner with bromine water and hypo.

(*b*) *Prussic Acid.*

efore applying any other test, I first use a strip of filter paper dipped first in a eak solution of copper sulphate, and then in freshly prepared tincture of guaiacum. (tartaric acid) distillate contains prussic acid, the paper when moistened with it rn blue. If no colouration is got, it is quite superfluous to try any other tests, obtained, it is as well to try and get the prussian blue, the sulphur and also the russide test. As regards the prussian blue test, I must emphatically warn against too much ferrous sulphate as otherwise a yellow colour will almost completely e the blue. If the amount of prussic acid is very small, a precipitate will some- take a long time to form. When applying the sulphur test, the great thing is to an excess of ammonium sulphide.

f prussic acid has been found, one must not neglect to test the residue in distilling or possible ferrocyanides.

quantitative estimation of the prussic acid is conducted as follows :—The dis- is, if necessary, acidified with nitric acid, and precipitated with silver nitrate. recipitate is collected on a weighed filter, washed, dried and weighed. To make he precipitate is ignited and the residual silver also weighed.

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ote by Translator. A solution of mecurous nitrate containing nitrous acid; first used by or Plugge for the detection of phenol and allied bodies.—L. de K.

(c) *Phosphorus.*

To observe very plainly the glowing of the phosphorus, I must recommend the use of the bulb-tube. The vapours for a long time condense in the bulb, which, in consequence, will appear quite luminous. If the phosphorescence vanishes, it may be several times made to again appear by opening the steam apparatus for a moment (and temporarily removing the flame) so as to let a little air get in. After distilling for about an hour, the distillation is stopped. All the while the receiver must have been kept very cool and also connected with a **U**-tube containing solution of silver nitrate. After particularly noticing whether there are any globules of phosphorus, the liquid may be tested for phosphoric acid by means of chlorine water and ammonium molybdate. A quantitative estimation may be attempted, but only about half the original phosphorus will, as a rule, be found. If no phosphorus has been detected, it must not be omitted to test the residue in retort for *phosphorous* acid, say by Dusart-Blondlot's process.

ALKALOIDS AND ALLIED BASES.

The testing of organs for alkaloids is, as is well known, one of the most difficult chemical problems, which fact will be admitted by every analyst of long standing and experience. Our imperfect knowledge of the ptomeines, and the conditions necessary to their formation and disappearance, makes it so difficult to judge about the alkaloidal nature of a substance. There has already been such an amount of literature about ptoma-conine, ptoma-strychnine, etc., that I should not wonder to notice one of these days a pamphlet on the preparation of strychnine, morphine, etc., from putrefying white of egg. It is argued with much truth, that a nitrogenous base, isolated from organs, is only then to be pronounced a vegetable alkaloid when it possesses *all its physical, physiological and chemical properties.* Of course, in such a case, there is no fear of its being a ptomeine; but alkaloids are generally present in such small quantities, that it is mostly impossible to get *all* reactions satisfactorily, and I feel sure that many a too-particular analyst has in consequence been induced to call vegetable alkaloids ptomeines.

It certainly is much easier, in the present state of science, to pronounce a residue to be a ptomeine instead of an alkaloid, for the simple reason that one rarely ever succeeds in isolating the base from the organs in *a perfectly pure condition.*

The best way to extract the alkaloids is by means of a alcoholic solution of tartaric acid. The fat which separates out scarcely deserves any further attention, although it is not quite impossible that it may act as a solvent for organic poisons;[*] but it mostly consists for the greater part of cholerestin. The extracts got from a stomach are mostly of a bright yellow colour, more particularly if there was an abundance of bile; but organs containing much blood give extracts of a much darker colour, and should therefore be analysed separately.

A clarification of the solution may be attempted by adding alcoholic solution of tartaric acid as long as a precipitate forms. The filtrate is evaporated, the residue taken up with water, and then nearly neutralised with potash. On adding alcohol, assium tartrate is precipitated, carrying down a good deal of the colouring

---

[*] For instance, nitroglycerine. In the absence of fatty matter, this is best isolated by extracting the air-dried substance with chloroform, finally drying the extract over sulphuric acid.

ter evaporating off the spirit, the watery fluid is once more tested as to its , and, if necessary, neutralised with potash. As it is difficult to get perfect ty, it is best to use very delicate litmus paper, working until only the acidity is noticed. Before using the regular course, one must never omit little of the solution for meconic acid, and also to notice its behaviour towards id. If no iodine is liberated (acidify the fluid with tartaric acid), it is no use for morphine; but of course a separation of iodine does not in the least e presence of that alkaloid, as other bodies may cause the reaction. The fluid ivided into three parts. Part 1 is made alkaline with potash and distilled in a of steam or hydrogen. The distillate is to be especially tested for nicotine, and aniline, which is done best by first shaking it with ether and evaporating current of air, the flask containing the solution being surrounded with water 80°C. When about 10 c.c. of ether are left, it is put to spontaneously evaporate ll beaker. The residue is dried in vacuo over sulphuric acid, and then made eutral hydrochloride.* If necessary, the usual tests for nicotine may be sup-ed by preparing the double salts with gold or platinic chloride, and sub- these to a quantitative analysis.

no volatile bases have been found, the second portion is shaken out: 1. With oth from acid and alkaline solution. 2. With chloroform from alkaline solu-. With amylic alcohol after addition of ammonium chloride, but this is only y if the iodic acid test has been successful. Before shaking with the chloroform, er must be expelled by gentle warming and blowing, and before using the amylic any chloroform must be likewise got rid of.

reactions are got which point to the presence of a particular alkaloid, a special is applied to the third part of the fluid. It is a good plan to add solution of c chloride, which precipitates every trace of nitrogenous base in twenty-four The deposit is washed with a weak solution of sublimate, and the alkaloid is lated in the usual manner. The residues left after evaporation of the ether, rm, and amylic alcohol must always be dried over sulphuric acid; they are y amorphous and crystalline, colourless, or of a yellow or brown colour. 's reagent is often reduced with a blue colour, to which, however, not much nce must be attached. The same may be said of the passing green colour with a sulphuric acid solution of ammonium vanadate. The residue from the ether will nearly always get violet on warming with syrupy phosphoric acid, h this test has been supposed to be characteristic for aconite only.† The other-splendid atropine test of Vitali (treating the pure alkaloid, first with fuming id, and then with alcoholic potash) will, however, mostly fail with the impure The same failure is sure to occur on applying the chromic acid test (getting eable odour).

---

nine hydrochloride is of characteristic appearance.

te by Translator. I have often seen syrupy phosphoric acid turning violet on heating, most used by some organic matter.

As a test for strychnine, ammonium vanadate has lately been strongly recommended and found trustworthy. The experiment is best performed in two ways. First of all, the solution of the vanadate in sulphuric acid is dropped on to the alkaloidal residue; and, secondly, solid vanadate is first added and the whole moistened with sulphuric acid. This test is preferable to the time-honoured test with bichromate, at least, when the alkaloid is somewhat coloured.

I must now call attention to an important fact, as far as I am aware, not yet published, viz.: Colocynth resin gives with vanadate solution a reaction almost like strychnine.

If this drastic (colocynth) is suspected, I proceed as follows:—The residue is mixed with a pinch of powdered bichromate with addition of a few drops of dilute sulphuric acid (1-2). At a little distance a little strong sulphuric acid is placed, and the two liquids drawn together with a glass rod. A beautiful violet-red is thus obtained, which gradually increases in colour. If vanadate is used instead of bichromate a blue colour is first obtained and a violet-red afterwards.*

The residue obtained by evaporation of the amylic alcohol (which must first be passed through a dry filter) is often impure, and is best purified by treatment with alcohol. The morphine is then tested for by the ordinary reagents, or it may be tested after conversion into apomorphine.

METALLIC POISONS.

I either use the residue from the distillation or the residue from the extraction with alcohol, in which case the latter is expelled by gentle heat. After adding boiling water and a sufficiency of potassium chlorate, hydrochloric acid is gradually added until the organic tissues are disintegrated. I am sorry to say I cannot recommend Jeserich's plan of using chloric acid, as this seems never to be quite free from arsenic, otherwise it is an excellent process. After warming for some time to expel chlorine, if necessary with an occasional addition of hot water, a little tartaric acid is added to keep up any antimony, and the whole set aside for twenty-four hours.

(*a*) *The insoluble portion.*

This is collected on a filter and washed with water until the washings are colourless (if there should be a large residue, it is a good plan to once more treat with chlorine), then with alcohol, and finally (in a beaker) with ether, until the fat is practically removed. The residue is now burnt in a porcelain dish. The ash is first digested with weak hydrochloric acid and the insoluble ash collected, washed, dried, ignited, and weighed. If it should not merely look like sandy matter, its nature must be investigated, best by fusing it with (previously ignited) sodium hydrocarbonate in a platinum crucible. After treating the mass in water, any insoluble matter is dissolved in nitric acid, and the solution tested with hydrochloric acid (silver), sulphuric acid (lead, barium, strontium), and hydrogen sulphide. The alkaline filtrate is mixed with excess of hydrochloric acid and evaporated to dryness (to render silica insoluble). After taking up with acid water and filtering, the liquid may be tested for alumina. In a case of poisoning by barium chloride, where sodium sulphate had been given as an antidote, I ould not find any barium in the stomach, œsophagus, and duodenum, but got as much s ·158 gram. of barium sulphate from the abdominal glands, brains, and lungs, whose oint weight was 719 grammes.

---

* Note by Translator. This is very nearly the same as with strychnine, showing, after all, the advisability of always using the chromate reaction when testing for strychnine.

(*b*) *The soluble portion.*

I must state that it is absolutely necessary the fluid shall contain no free chlorine. fter making up to a definite bulk, say 500 c.c., aliquot parts are taken and submitted o analysis.

(*a*)

Fifty to 100 c.c. are nearly neutralised with caustic potash in a porcelain dish, a mall coil of clean brass is introduced, and the whole warmed for fifteen minutes. The oil is then washed with water, alcohol, and ether, and after drying (in desiccator) ntroduced in a combustion tube partly filled with coarse oxide of copper, and drawn ut so as to be connected with two capillary tubes. By means of an aspirator a slow urrent of dried air is drawn through and the tube heated as if an elementary analysis ere conducted. Any mercury will condense inside the capillary tubes.

Whilst still hot a drop of water is put on the combustion tube (close to the apillary one) and is so made to break. A minute particle of iodine is now introduced, nd with the application of a gentle heat, its vapour mixed with the air current, is ade to act on the mercury globules, which will then cause the formation of the haracteristic mercuric iodide. As regards this very reliable process, I want to bserve that a long immersion of the brass is not advisable because it gets too thickly oated with organic matter.

The quantitative estimation of the mercury I conduct as follows:—Through the ot acid solution, I pass hydrogen sulphide, and collect the precipitate on an asbestos filter. fter washing with strong hot hydrochloric acid, the asbestos is rinsed into a beaker nd digested with a few drops of nitric acid, which will soon dissolve out the mercury. fter filtering through the same asbestos the mercury is precipitated as mercurous hloride by the addition of excess of phosphorous acid, which is allowed to act for twenty-our hours.

(*b*) *Antimony.*

Fifty c.c. of the liquid are put into a clean platinum dish, the excess of acid neutralised ith ammonia, and a piece of pure zinc is introduced. After acting for six hours, the iquid is poured off and the antimony will have blackened the platinum, or, if much was resent, deposited itself as a spongy mass. The liquid may contain traces of it, as antimony is somewhat soluble in concentrated solution of zinc chloride. Its quantitative etermination is conducted as follows:—Though the hot fluid I pass hydrogen sulphide ntil the liquid has got cold. After standing for three days, the excess of hydrogen ulphide is removed by a rapid current of carbonic acid, a little ether being, if necessary, dded to prevent frothing. The precipitate is filtered off and washed with a weak solution of ammonium acetate. It is then rinsed from the filter with solution of caustic oda, which is then saturated with hydrogen sulphide, which will cause the antimony to issolve. After filtering off from any insoluble matter, the liquid is slightly acidified ith hydrochloric acid and heated to boiling. After standing for a day, the precipitate filtered off, washed, and finally oxidised with nitric acid. To completely get rid of organic matter, the mass is made alkaline with caustic soda, dried and fused with a mi of nitrate and carbonate of soda. The mass is now exhausted with proof-spirit, an insoluble sodium antimoniate washed with proof-spirit to which a few drops of so carbonate have been added. It is then digested in a mixture of hydrochloric and ta acids, the liquid nearly neutralised with ammonia, and once more precipitated hydrogen sulphide. The precipitate, which should now possess the characteristic of antimony sulphide, is not weighed as such, but I prefer to make it into oxi treatment with fuming nitric acid, taking all precautions as advised by Bunsen.

To make sure of the absence of tin, I place the uncovered crucible in the redu flame of a powerful burner, when the antimony will gradually, but completely vola

(To be continued.)

METHODS FOR DETECTING OLEO IN BUTTER AND COTTON SEED IN LARD.

BY THOMAS TAYLOR, M.D., MICROSCOPIST, UNITED STATES DEPARTMENT OF AGRICULTURE.

Abstract of paper read before the Chemical Society of Washington, D.C., March 1890.

TO DETECT OLEO IN BUTTER.

[I believe that this method will prove useful for all practical purposes unde oleomargarine laws of the United States.]

Dissolve in 20 c.c. of petroleum benzine 140 grains of a mixture of oleo butter. Heat slightly to secure a perfect solution of the fats. Caseine and a tissues may be removed by filtering the liquid while it is warm. Fill a test- with the filtered solution and place it in ice-water. In from five to tw minutes the oleo fat will separate from the butter fat, and falls to the botto the tube, being sparingly soluble in cold benzine, while the butter fat remain solution in cold benzine. Separate the oleo fat from the liquid butter by filtra The fat recovered may be solidified by mechanical pressure, placing it bet several layers of filtering paper to absorb the remaining benzine, after which sheet of solid oleo may be removed from the paper with a palette-knife. The bu may be recovered by evaporating the benzine by means of heat.

TO DETECT COTTON SEED OIL IN LARD.

Dissolve in 20 c.c. of petroleum benzine 140 grains of a mixture of lard cotton seed oil. Heat slightly to secure a perfect solution of the lard. Rem animal tissues by filtering as above. Fill a test-tube with the filtered solution place it in ice-water. The fat will be precipitated, and falls to the bottom of tube by reason of its comparative insolubility in cold benzine, while the cotton oil remains in solution. Separate the lard from the cotton seed oil by the us filtering paper, and subject the recovered fat to pressure as in the case of bu and oleo, by which means the remaining benzine is absorbed. The solidified fat ma removed from the paper with a palette-knife. The benzine is separated from the co seed oil by means of heat.

ELECTROLYTIC SEPARATIONS.

By Edgar F. Smith and Lee K. Frankel.

(*Continued from page* 78.)

MERCURY FROM NICKEL.

With these two metals the current was allowed to act for sixteen hours. The sults are as follows :—

	Mercury present in grams.	Nickel present.	KCN in grams.	Total dilution.	Current strength in c.c. O-H gas per minute.	Mercury found.	Difference in per cent. from the theoretical.
1	0·2440	100 per ct.	$4\frac{1}{2}$	200 c.c.	0·4 c.c.	0·2435	−0·20 per cent.
2	„	„	„	„	„	0·2432	−0·32 „
3	„	„	„	„	„	0·2425	−0·60 „

MERCURY FROM COBALT.

Our experience with these two metals was so unexpected that we append the poor well as the good results which were finally obtained :—

	Mercury present in grams.	Cobalt present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Mercury found.	Difference in per cent. from the theoretical.
1	0·2440	100 per ct.	$4\frac{1}{2}$	200 c.c.	0·4 c.c.	0·2386	−2·21 per cent.
2	„	„	„	„	„	0·2388	−2·13 „
3	„	„	„	„	„	0·2364	−3·11 „
4	„	„	„	„	„	0·2333	−4·00 „
5	„	„	„	„	„	0·2366	−3·02 „

The current acted for sixteen hours upon reducing the quantity of cobalt, and erating with the conditions, in other respects, the same as before (except in 11 and), we obtained :—

	Mercury present in grams.	Cobalt present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Mercury found	Difference in per cent from the theoretical.
6	0·2440	75 per ct.	$4\frac{1}{2}$	200 c.c.	0·5 c.c.	0·2353	−3·56 per cent.
7	„	„	„	„	0·86 c.c.	0·2357	−3·40 „
8	„	50 per ct.	„	„	0·5 c.c.	0·2387	−2·25 „
9	„	„	„	„	„	0·2381	−2·33 „
0	„	„	„	„	0·86 c.c.	0·2321	−4·80 „
1	„	25 per ct.	3	„	0·5 c.c.	0·2442	+0·09 „
2	„	„	$4\frac{2}{1}$	„	0·86 c.c.	0·2342	−4·01 „
3	„	$12\frac{1}{2}$ per ct.	3	„	0·5 c.c.	0·2445	+0·20 „

Inspection of these figures discloses the fact that nothing approaching a separation of the two metals appears probable until in experiment (11), where not only the quantity of cobalt is reduced, but also that of the potassium cyanide. The result is then surprisingly close (+ ·09 per cent.). This would seem to be due rather to the reduction of the quantity of cyanide, inasmuch as by its increase in experiment (12), we again have a minus error of 4·01 per cent., and in experiment (13), by reducing the quantity of cyanide to 3 grams., the result is satisfactory. As the quantity of cobalt in this instance was but half of that in experiment (12), the favourable result might be attributed to this. Hence the following trials were made :—

	Mercury present in grams.	Cobalt present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Mercury found.	Time of precipitation.	Difference in per cent.
14	0·2250	—	3	200 c.c.	0·5 c.c.	0·2250	16 hrs.	—
15	”	25 per ct.	”	”	”	0·2256	”	+0·26 per ct.
16	”	50 ”	”	”	”	0·2258	”	+0·35 ”
17	”	75 ”	”	”	”	0·2258	”	+0·35 ”
18	”	100 ”	2·2	”	”	0·2235	”	−0·60 ”

Cobalt was not found in the mercury deposit, nor mercury in the cobalt solution.

Evidently the quantity of cyanide present exercises a marked influence upon the separation. Returning to the separation of cadmium from cobalt, it will be observed that the amount of cyanide present there was 4½ grams., while the quantity of metal was less; yet the separation proved satisfactory.

SILVER FROM COPPER.

It yet remains for us to record some experiments upon the separation of these two metals in cyanide solution. Reference to a former paper (*American Chemical Journal*, 11, 264, and *Journal of Analytical Chemistry*, 3, 254) will show that our attempts in this direction were at that time fruitless. It was after the successful separation of cadmium from copper in cyanide solution that we were impressed with the idea that the separation of silver from copper ought to occur, since silver deposits so readily, even when exposed to a very feeble current. In the communication to which we refer, the current strength recorded was 1 c c. O-H gas per minute. Since it was by carefully reducing this in other cases that we obtained good separations, we instituted a new series of experiments with silver and copper, acting upon the mixture with a much weaker current :—

	Silver present in grams.	Copper present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Time in hours.	Silver found.	Difference in per cent.
1	0·1788	—	4½	200 c.c.	0·10 c.c.	16	0·1792	+0·22 per ct.
2	”	—	”	”	0·23 ”	”	0·1788	—
3	”	100 p. c.	”	”	0·15 ”	”	0·1788	—
4	”	”	”	”	0·30 ”	”	0·1787	− 0·05 per ct.
5	”	”	”	”	0·20 ”	”	0·1784	− 0·20 ”
6	”	”	”	”	” ”	”	0·1782	− 0·33 ”
7	”	”	”	”	0·40 ”	”	0·1783	− 0·28 ”
8	”	”	”	”	0·15 ”	”	0·1800	+0·60 ”

next dissolved 0·1732 gram. pure metallic silver in nitric acid. To its solu- r evaporation, there were added 100 per cent. copper and 4½ grams. potassium On electrolysing, with a current of 0·15 c.c. O-H gas per minute, the resulting eighed 0·1725 grams. The latter contained no copper, nor could silver be in the copper solution.

ilver ten-cent piece, weighing 1·2236 grams., was brought into solution and o 100 c.c. Of this solution two portions (25 c.c. each) were electrolysed in the of 4½ grams. potassium cyanide, with a current of 0·4 c.c. O-H gas per minute. r found was in—

(*a*) 89·64 per cent.
(*b*) 89·56 per cent.

silver determinations made with another coin gave :—

(*c*) 89·44 per cent.
(*d*) 89·44 per cent.

(*To be continued.*)

LAW NOTES.

Convictions for the Same Sample of Milk.—Mr. B. S. Weston, Sanitary Inspector tthew, Bethnal Green, submitted a sample of milk purchased from the Farmers' Direct sociation to the Public Analyst, Mr. A. W. Stokes, F.I.C. A certificate was returned stating ontained 10 per cent. of added water, and that 33 per cent. of the cream had been abstracted ample. Two summonses were taken out, one under section 6 and one under section 9 of ration Act. On February 28th, at Worship Street, Mr. Bushby heard the case. The defen- red by his Solicitor (Mr. Young), but the case was given against him, and a fine of £5 was r the abstraction of the cream and £2 for the addition of water, costs being given in both

REVIEWS, NOTES, Etc.

rnal of the Royal Agricultural Society.—We have received the first f the new series (the third) of this journal. It is to be issued quarterly, and ll printed and containing much that is vitally important to all agriculturists, it rtainly be appreciated by all interested in the subject. We observe very inter- ports from the society's consulting naturalist, and also from the botanist; while st exposes a series of frauds in feeding meals, adulterated samples of which e very prevalent of late.

s in Practical Chemistry; an Introduction to Qualitative and ntitative Analysis, by Dr. W. R. Hodgkinson, F.R.S.E. London: George nning, Great Queen Street.

little brochure in pamphlet form of 64 pages, giving the usual reactions and he various metals and acids, and a few of the gases, with tables for quantitative It possesses no special feature, but is well and concisely put. Like many its class, it is, in our opinion, sorely deficient in a really systematic acid course, being left to the student's initiative in this matter. So far as qualitative work roduction to quantitative, then the title is correct, but we have looked in vain ther attempt to explain quantitative analysis.

Sugar Reagent.—Matthieu-Plessy recommends the following as a reagent for cane-sugar, grape-sugar, and pyrogallic acid: 54 pts. of ammonium nitrate are fused, and 34 pts. lead nitrate and 21 pts. of plumbic hydrate are added. The reagent melts at 105° C., and with the substances named gives mixtures, having in the presence of glucose a cherry red, with cane-sugar a coffee-coloured, and with pyrogallic acid a chrome-green colour.—*Am. Journ. Pharm.*

Chemically Pure Sulphate of Quinine may be distinguished from the commercial sulphate and the sulphates of cinchonine, cinchonidine and quinidine by a solubility test in a mixture of chloroform and petroleum ether. 0·2 gram. are briskly agitated with a mixture of 30 parts by volume of petroleum ether (sp. gr. 0·680) and 70 parts chloroform, filtering and adding 3 to 4 volumes of petroleum ether; in the absence of the the other sulphates the solution will remain clear. An admixture of only 0·1 per cent. other sulphate will give rise to a precipitate or turbidity.—(E. Hirschsohn.)

Salt Regal.—This attractive effervescent saline, which has attained a high reputation on the Continent, the Colonies, and India, is, we are informed, about to be energetically pushed in this country. It should do well, because it is an original idea, and has been certified by Dr. Paul and several other analysts as being perfectly pure in all its ingredients. It is the only attempt that has yet been successfully made to combine an aperient with an active oxidising disinfectant without decomposing the latter, and is, therefore, chemically interesting to that extent.

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THE ANALYST.

JUNE, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

GENERAL meeting of the Society was held at Burlington House, on Wednesday 14th t., the President, Mr. Adams, in the chair.

The minutes of the last meeting were read and confirmed.

On the ballot-papers being opened it was announced that Mr. W. Droop Richmond as elected as a member.

The following gentlemen were proposed for election :—As member, Mr. S. J. Steel, hemist to Messrs. Martineau and Sons. As associate, Mr. T. H. Pearman, Assistant Mr. A. H. Allen.

The following paper was read by the President, "Remarks on the Bitter of the op as Differing from the Bitter of Quassia and other Hop Substitutes."

The reading of the paper and the subsequent disscusion extending to a late hour, e other papers announced to be read were necessarily postponed.

The next meeting of the Society will be held at Burlington House on the 11th inst.

(*Conclusion of the Society's Proceedings.*)

ON THE ANALYSIS OF PEPTONES.

BY M. ALPHONSE DENAEYER, BRUSSELS.*

T the last meeting of the Association I had the opportunity, gentlemen, of drawing our attention to the analogies of reaction which peptone and gelatine present; and I nsisted on the fact that in the analysis of the peptones of commerce, the gelatine, hich in the meat peptones is due to the transformation of the tendinous parts of the muscular tissue, was precipitated by phospho-tungstic acid, as well as the real peptone of albumen and the albumose. The gelatine contains nitrogen, and therefore in the application of the König process for the determination of peptones, the gelatine is inevitably precipitated as well as the peptone, and the estimation of nitrogen under these conditions gives an important excess of peptone.

---

* *Bulletin de l'Association Belge des Chimists*, March 31, 1890.

M. Bruylants said in the last discussion we had on the question, that the double iodide of potassium and mercury which I showed to be distinctive of albumose, peptone, and gelatine, was perhaps the basis of a system of separation of these bodies. The late hour of the meeting prevented my presenting to you the whole results of my researches on this point, and the application which I had already made of the reagent in the separation of these compounds. Keeping in mind the ideas I have expressed as to basing the operations of estimation on the rational preliminary separation of gelatine, albumose, and peptone, I use for the analysis of preparations of peptones the technical processes which I am about to show you. They are different from those suggested by König, but I think that elements for discussion and experiment will be found in them which will lead, perhaps, to new improvements.

METHOD FOR THE ANALYSIS OF PEPTONE PREPARATIONS.

The composition of these preparations always approximates to that of the albuminoids from which they have been derived by the action of the gastric juice. The constituent elements which it is necessary to determine in the analysis of peptones are the following:—1. Water. 2. Ash. 3. Insoluble matters, if any. 4. Gelatine. 5. Albumose. 6. Peptone. 7. Definite compounds. Among these elements we must remark that insoluble gelatine and the defined compounds may be absent.

Water.—The proportion per cent. is determined by evaporating in a water-bath some grammes of peptone. The complete desiccation is continued in an oven at 100° C. The residue is weighed after standing in vacuo over concentrated sulphuric acid for twenty-four hours.

Ash.—The residue previously dried is ignited and weighed accurately.

Insoluble matter.—The peptone is diluted with water, the insoluble residue is collected on a tared filter, washed with water, dried in the oven at 100° C., and weighed after cooling under the desiccator.

Gelatine.—The estimation is founded on its precipitation by ammonium sulphate; but as the albumose is also precipitated by this reagent, it is necessary, as a preliminary, to separate this compound. For this purpose I employ iodide of potassium and mercury, using Mayer's formula:—

Iodide of potassium	49·801 grammes.
Chloride of mercury	13·546 "
Distilled water	1,000·000 "

A known weight of peptone, 1 or 2 grammes, dissolved in water, is precipitated by an excess of the reagent. After filtration and washing, the liquors are evaporated to a few cubic centimetres, then an excess of ammonium sulphate in saturated solution is added. The mixture is brought to the boiling point, which causes the coagulation of the gelatine against the sides of the vessel. When quite cold, the liquid is decanted, and the gelatine washed first with ammonium sulphate, then rapidly with cold water, and finally with boiling alcohol, which removes the double iodide of potassium and mercury entangled by the precipitate. The gelatine remains adhering to the glass; the vessel is then dried in the oven and weighed. The gelatine still retains some ammonium sulphate, *which it is necessary to deduct from the weight obtained.* For this purpose the coagulum is dissolved in warm water, an excess of hydrochloric acid is added, it is filtered, and the filter washed with as little distilled water as possible. The ammonium sulphate is titrated in solution by a normal solution of chloride of barium, the end of the reaction being ascertained by filtering small portions to which drops of barium chloride are added.

One c.c. of normal chloride of barium containing $\frac{120}{1000}$ of crystallised salt represents 0·08 gramme of ammonium sulphate.

Albumose.—One or two grammes of peptone are taken and dissolved in 5 c.c. of distilled water. An excess of ammonium sulphate is added to the clear solution. This reagent precipitates the albumose and gelatine if any are present. The precipitate is collected on a tarred filter. This is washed with solution of ammonium sulphate, dried and weighed. The precipitate is then dissolved in a sufficient quantity of hot water, and after filtration the ammonium sulphate is estimated by the process described above. From the weight of the precipitate obtained is deducted (1) the weight of the gelatine; (2) the weight of the ammonium sulphate; the difference giving the weight of the albumose.

Peptone.—One or two grammes of peptone are taken as before. The peptone is then dried in vacuo at 60° C., which produces great inflation of the mass. The porous residue is washed with alcohol at 95° in order to eliminate creatine, creatinine urea, sarcolactic, formic, acetic and butyric acids, which are especially found in peptones prepared directly from meat. These compounds can be estimated in mass by evaporation of the alcohol. The residue is taken up by some grammes of water, and an excess of a solution of phosphotungstate of soda thus prepared is added

Tungstate of soda	50 grammes.
Boiling distilled water	1000 „
Phosphoric acid (medicinal)	100 „
Pure hydrochloric acid	150 „

This last acid must be added after the solution is cold. The liquid is filtered after standing for twenty-four hours.

This reagent precipitates the gelatine, the albumose and the peptone. After settlement a small portion of the clear liquid is tested, then the greater part of the solution is decanted and the precipitate is thrown on a Schleicher's filter carefully tared. The precipitate is dried in the oven, weighed after cooling in the desiccator, and the weight noted.

The precipitate is then introduced into a porcelain capsule and ignited in a gas muffle to total incineration of the organic matter and filter. This last residue is weighed and its weight, minus the weight of the filter ash, is deducted from that of the dry precipitate. The difference represents the weight of the peptonic matter precipitated. From this is deducted (1) the weight of the gelatine; (2) the weight of the albumose. The last difference gives the weight of the peptone.

This method gives results as accurate as those based on the determination of peptone by the estimation of nitrogen, and it has the advantage of being much quicker. Comparative analyses of eight samples of different peptones have given results differing but slightly from those obtained by calcination.

Definite Compounds.—Under this denomination are included the nitrogenous bases and the fatty and amido acids of muscular tissue.

The definite compounds are divided into nitrogenous and non-nitrogenous substances. Some are soluble and others insoluble in alcohol. Taking this classification I group them in the following order: Substances soluble in alcohol:

Nitrogenous : Creatine, creatinine urea.
Non-nitrogenous : Sarcolactic, formic, butyric, and acetic acids.

These substances may easily be determined collectively by evaporation of the alcoholic solution obtained by washing the product yielded by evaporation to dryness in vacuo before the precipitation of the peptone by phosphotungstate of soda. Substances insoluble in alcohol:

Nitrogenous : Taurine.
Non-nitrogenous : Inosite, dextrin, glycogen.

The proportion of these elements in the preparation of the peptone analysed is determined by difference.

I have applied my method of analysis to several commercial preparations of peptone as well as to the peptones specially prepared by myself. It is executed with perfect regularity. The following are three results selected from among the numerous applications which I have made of the method. They bear on the principal types of peptone preparations.

Designation.	Peptone of purified meat-albumen.	Peptone of egg-albumen.	Peptone direct from meat.
Peptone..	37·675	34·700	25·857
Albumose	31·300	53·350	15·964
Gelatine	none	none	9·826
Definite Compounds	5·525	5·930	29·972
Mineral Salts	8·285	1·025	18·386
Water	10·250	4·625	none
Insoluble	9·965	3·980	none

These figures mark profound differences of composition in the relative proportions of peptone and albumose. The peptone of meat albumen and the direct peptone of meat do not offer a constant ratio in regard to the proportional figures of albumose.

This anomaly may be imputed to the mode of preparation employed, which is more efficacious for the peptone of meat albumen than for that of coagulated albumen, which last is less easily peptonisable than albumen in solution in the preparation of meat peptone properly so called.

A METHOD FOR THE DETECTION AND ESTIMATION OF PETROLEUM IN OIL OF TURPENTINE.

By W. M. Burton, Cleveland, Ohio.*

The use of the lighter boiling products of petroleum for the purpose of adulterating oil of turpentine is so well known to persons using these articles that little comment is necessary concerning the prevalence of this adulteration. Refined petroleum of the proper colour and gravity can be mixed with turpentine in such proportions as to elude the common methods of detection. The gravity, colour, and odour of the mixture can be made identical with that of turpentine; whereas paint made from such material is not durable, but is liable to crack and peel off soon after its application. The pecuniary advantage to the unscrupulous dealer is also a great temptation to perpetrate this adulteration on the large scale. The method which is here proposed is based upon the fact that petroleum is only slightly affected by fuming nitric acid in the cold, whereas turpentine is readily oxidised to various acids of the fatty and aromatic series, which are soluble in hot water. If, therefore, we treat a mixture of turpentine and refined petroleum with cold fuming nitric acid, the turpenes will all be converted into acids readily soluble in hot water, while the paraffins of the petroleum will remain almost entirely unaffected. These facts enable us to determine almost quantatively the amount of petroleum contained in adulterated turpentine. The apparatus necessary for effecting this separation is very simple. A balloon flask of 750 c. cm. capacity is fitted with a two-hole cork stopper. Through one hole is inserted the tube of a glass stoppered drop funnel having a capacity of 100 c. cm. The flask is also connected with an inverted condenser. About 300 c. cm. fuming nitric acid of sp. gr. 1·4 are placed in the flask, and 100 c. cm. of the turpentine to be tested are measured into the drop-funnel. The flask is surrounded by cold water, and the turpentine is allowed to drop slowly into the nitric acid. As each drop strikes the acid, violent action takes place, with evolution of red fumes. It is well to shake the flask occasionally during the operation. When the turpentine has all passed into the flask, the apparatus is allowed to stand until all action is over. The contents of the flask are transferred to a large separating funnel and treated with successive portions of hot water. In this way all the products resulting from the action of the acid on the terpenes are removed, while any petroleum paraffins remain insoluble in water, and can readily be separated and measured.

Having procured some turpentine known to be free from petroleum, my assistant prepared mixtures of the pure turpentine with samples of refined petroleum of various boiling points. In order that my judgment in experimenting might not be influenced by knowing the quantities of material used, my assistant withheld from me the proportions employed in the mixtures. Ten mixtures were analysed, with the following results:—

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* *School of Mines Quarterly.*

Pure turpentine used.	Petroleum used.	Boiling point of petroleum.	Petroleum found by method described.
66 c. cm.	35 c. cm.	about 250°	34·1 c. cm.
80	20	250	18·9
70	30	200	29·
80	20	200	18·5
90	10	200	8·9
80	20	100	17·8
70	30	100	28·4
85	15	100	13·5
80	20	75	17·9
70	30	75	28·

It will be noticed that the sum of the amount of petroleum and turpentine used would be 100 c.cm. in each case, so that the number of cubic centimetres of petroleum found would also express the percentage of adulterant. It appears from the above results that the petroleum fractions, which boil at about 250° are least affected by the fuming nitric acid, while the low-boiling fractions are affected the most. But in order to keep the specific gravity of the turpentine where it should be, the higher boiling petroleum fractions must be used in the adulteration— a fact which is favourable for the application of this method in the analysis of such mixtures.

A SIMPLE METHOD FOR ESTIMATING UREA.

By C. W. Heaton, F.I.C., F.C.S., and S. A. Vasey, A.I.C., F.C.S.*

Several years ago it was suggested to us by some of our former pupils that it would be convenient if a system could be devised by which urea could be estimated easily, rapidly, and accurately without specially constructed apparatus. The hypobromite method gives results which are quite correct enough for clinical purposes, and several forms of apparatus of great convenience and simplicity have been devised for its execution. The latest and perhaps the simplest is that of Doremus, recently advocated by Dr. Cruise, of Dublin.

There is no difficulty in using any of the instruments which have been recommended, and it is probable that physicians will prefer to employ some one of them when it is accessible. But such instruments are not always accessible, and are easily broken; and it is in any case desirable that the medical practitioner who chances to have no apparatus at his command except the ordinary weights and measures of pharmacy, shall be able, without trouble or loss of time, to make satisfactory estimations of urea. In the system arranged by us in 1884, and now for the first time described in print, absolute accuracy is not aimed at, but the maximum error of calculation is so minute as to be quite lost in the greater errors due to variations of temperature and pressure, and erfect measurement which are incidental to any rapid process which depends on easurement of the nitrogen and expelled by alkaline hypobromite. It is well that this nitrogen amounts approximately to 92 per cent. of the total nitrogen of The apparatus required is of the simplest character. In addition to the ordinary aceutical measures, all that is really necessary is a thistle-headed acid funnel, about ot of glass tubing, and a couple of bottles. It is well to assume that no other nces are available. The reagents are bromine and caustic soda.

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* *The Lancet*, May 10th, 1890.

he following arrangements are made :—1. An eight-ounce bottle of any form is with a thistle-funnel and bent glass delivery tube, as though for the preparation rogen. The lower end of the funnel should be bent upwards, like a small hook, vent gas from passing up it. 2. A small basin or breakfast cup may be used as a atic trough, a four-ounce, or larger, bottle of any form being filled with water and ed in it in such a manner that the end of the gas delivery tube can readily be it under the mouth of the bottle. 3. A 40 per cent. solution of good commercial soda is prepared. For example, half a pound avoirdupois of soda may be dis- in water and when cold diluted to one pint.

he analytical process is as follows :—1. Into the gas generator is poured by means funnel one fluid drachm of bromine washed in by ten fluid drachms of the soda n. The generator may then be immersed in cold water, and the inverted bottle er placed over the end of the delivery tube. 2. Two fluid drachms of urine, very ly measured, are added and washed in by exactly one fluid drachm of water. The luid drachms so added will of course cause an equal volume of air to pass into the ng bottle. This is allowed for in the appended table. The generator is gently ; brisk effervescence takes place, and gas equal in volume to the liberated n is collected in the receiver. The generator should be kept as nearly as possible temperature of the air. 3. When the evolution of gas ceases, the receiver is ed from the basin by means of the thumb or a glass plate and placed mouth up- on the table. It is now only necessary to measure in minims the quantity of required to fill it. After deducting 180 (which may be taken as 200) minims the air displaced by the urine, each 100 minims of water added represent 0·25 nt. of urea in the urine examined. If the urine contains more than 3 per cent. of t is best to dilute it with an equal volume of water before making the deter- on.

he following table gives the percentage of urea corresponding to the volume of erated, as shown by the quantity of water required to fill the bottle :—

Minims of water required.	Percentage of urea.	Minims of water required.	Percentage of urea.
200	0·00	900	1·75
300	0·25	1000	2·00
400	0·50	1100	2·25
500	0·75	1200	2·50
600	1·00	1300	2·75
700	1·25	1400	3·00
800	1·50		

DETECTION AND QUANTITATIVE ESTIMATION OF ORGANIC AND INORGANIC POISONS IN BODIES.

By Dr. Anton Seyda.

(*Continued from page* 96.)

(c.) Detection and Estimation of Arsenic.

If no mercury or antimony are present, 50 or 100 c.c. of the liquid are put into a Marsh's apparatus and tested for arsenic. The zinc must be first put into hydrochloric acid for ten minutes, and then washed with water, in order to free it from traces of adhering arsenic. Of course before lighting the hydrogen or heating the reduction tube, one must wait until all air has been expelled. The fluid must be introduced in small quantities, otherwise there may be too much frothing. It is as well not to try and get arsenical spots, as these only form well when there is a rapid evolution of hydrogen and a large quantity of arsenic; but it is best to produce the arsenical mirrors, which may afterwards be sealed if necessary, and produced as evidence in court.

If the amount of arsenic was not too small, and one has succeeded in getting at least two mirrors, the tube is cut with a diamond in such a way as to get four pieces of the mirror. The following tests are then applied: One piece is heated and the arsenical odour observed. The second piece is moistened with sodium hypochlorite, when the mirror should of course disappear. The third piece is treated with nitric acid, and, after evaporating the acid, the residue is tested with silver nitrate for arsenious or arsenic acid. The fourth piece is heated with ammonium mono-sulphide, and the arsenic made into the yellow sulphide. These four tests are amply sufficient.

If arsenic has been found, it is quantitatively estimated as follows: The liquid is warmed and treated for twelve hours with hydrogen sulphide, then put aside in closed flask for about five days, until the precipitate has completely settled. After removing the excess of hydrogen sulphide by a current of carbonic acid, the precipitate is filtered off and washed. The filtrate, as a rule, gets turbid, but one need not pay much attention to this, as the cloudiness *may* be due to a trace of arsenic, but is generally caused by organic matter.

To purify the precipitate, it is best to use a solution of ammonium carbonate, which will leave at least the bulk of the free sulphur behind. After evaporating the solution, the residue must be treated with nitric acid, the solution evaporated and the product fused, best in a porcelain crucible, with a mixture of nitre and potassium—sodium carbonate. After dissolving the mass in water, it is best to slightly acidify with nitric acid, and after completely removing the carbonic acid and nitrous acids by boiling, once more to throw down with hydrogen sulphide. The precipitate must then again be made into arsenic acid,[*] and the solution, after concentration, be precipitated with magnesia mixture. To make sure of its purity, it is as well to redissolve the precipitate after slight washing with dilute ammonia, in hydrochloric acid, and once more throw down with ammonia. I have adopted with great success the proposal of de Koninck,[†] who dissolves the arsenical precipitate of the filter by means of nitric acid, evaporates the solution, and gradually heats the residue to redness. The pyroarseniate may be kept, or if there is not much of it, may be made into mirrors of metallic arsenic by means of the Marsh's apparatus, in the way already described.

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[*] Note by translator. Very conveniently done with bromine water.

[†] See Analyst, 1888, p. 178.

(*d.*) Tests for Other Metals, Tin Excepted.

If mercury, antimony, and arsenic are absent, the fluid is made alkaline with *pure* sodium hydrate, then acidified with acetic acid, and, after heated to boiling, treated with hydrogen sulphide until it is cold. Sodium carbonate is now added until alkaline reaction, and the corked flask set aside until the liquid has become quite clear. After collecting the precipitate (which, as a rule, consists mainly of ferrous sulphide) on a filter, it is washed with solution of sodium hydrogen sulphide until the washings run off colourless. The filtrate is kept for further investigation. The precipitate is now put into a porcelain dish, cautiously oxidised with nitric acid, and the acid evaporated off. The residue, after being moistened with a drop of strong soda, is mixed with a little dry sodium carbonate and put by degrees into a silver crucible containing fusing nitre. After cooling, the fused mass is exhausted with water, the alkaline fluid saturated with hydrochloric acid (in a beaker), and filtered off from traces of silver chloride through filter paper, but if permanganate should be present, through asbestos, the liquid is then qualitatively and, at the same time, quantitatively analysed by the usual methods.

About the metals, which are not precipitated by hydrogen sulphide from their acid solutions, I want to make some observations. As a rule, the separation of iron, zinc, and aluminium in presence of phosphates of lime and magnesia will have to be carried out. I proceed as follows: The hydrochloric solution of the sulphides is, after evaporation, mixed with some chlorine water and again concentrated. The residue is treated with slight excess of ammonia, which throws down the phosphates, the filtrate is acidified with acetic acid, heated to boiling, the zinc thrown down with hydrogen sulphide, and the zinc sulphide collected on a weighed filter. The phosphatic precipitate, which may contain alumina, is dissolved in nitric acid and heated in a platinum dish with metallic tin, which will cause the separation of the phosphoric acid as stannic phosphate. The filtrate is precipitated with ammonia, and the precipitate, after washing, burnt together with the filter. The ash is then fused with caustic soda for half an hour, which will form aluminate of soda, from which the pure alumina may be isolated in the usual way, after first extracting the alkaline mass with water, and filtering from any insoluble matter.

(*e.*) Estimation of Tin.

The sodium sulphide solution is acidified with hydrochloric acid boiled and treated with hydrogen sulphide. After twenty-four hours the precipitate is filtered off and washed with solution of ammonium acetate. The filter and contents is then heated in a porcelain crucible until the sulphide of tin has been converted into oxide, which oxidation may be assisted by the use of nitric acid. As, however, the oxide may contain traces of iron, it is best to fuse it in a silver crucible with pure caustic soda (free from water) for half an hour, at a temperature just high enough to melt the soda. After exhausting the mass with water, the fluid is filtered, acidified with hydrochloric acid and the tin precipitated with hydrogen sulphide. The stannic sulphide is then converted into oxide and finally weighed.

Final Remarks.

Among the normally occurring metals of the human body must be reckoned potassium, sodium, calcium, magnesium, iron, and manganesium. But one is sure to find also, aluminium, copper, zinc; even tin and lead. Of the last four there is, however, seldom more than one centigram. in a full-grown body. Traces of these metals of course get into the organism through the use of contaminated food, or impure beer, etc. If more than a trace is found inquiries should be made whether the deceased has been habitually taking medicines containing doses of, say, mercury or lead, or even, arsenic. As regards alumina (considering this is a constituent of sandy matter, clay, dust), traces of this substances may even get into the organs during the post-mortem examination, which, as we all know, is sometimes not too neatly conducted. As is well known, the organic matter rendered soluble by the treatment with chlorine is precipitated by hydrogen sulphide both in acid or alkaline solution. The removal of this organic matter by fusion with nitre answers very well for arsenic, antimony, and tin, but suppose there were mercury, this would volatilise. As I, however, test for mercury first by a special process, there is no danger of not finding it.

Contrary to the usual plan, I test for arsenic directly in the acid fluid, and only *then* proceed in the conventional way when the organs contain decided traces of free nitric acid, mercury, or aniline colours. Notwithstanding Otto's objections, I prefer this direct way of testing, which in fact had already been recommended by Marsh himself. It saves the troublesome treatment with hydrogen sulphide, which substance has lately become somewhat notorious as a possible source of arsenic.

To ascertain the influence of nitrates, I carried out the following experiment:—From a weak hydrochloric acid solution of ·75 per cent. nitre and ·33 per cent. arsenious acid, I added 1 c.c. to portions of organs, which were mixed up with water containing 5 grms. of potassium chlorate. After treating with acid as described, and freeing the filtrate completely from chlorine, the liquid was tested in the Marsh's apparatus, in which hydrogen was evolved from zinc and hydrochloric acid. After eight hours, practically all the arsenic had deposited in the reduction tubes. I must observe that addition of sugar seemed to favour the reaction.

From this experiment it seems that the presence of chlorides, and even nitrates, does not interfere with the formation of gaseous arsenic hydride, at least when there is plenty of zinc and acid, and sufficient time is allowed. Besides, as nitre speedily passes into the urine, no quantity of nitrate to speak of will ever be present in the organs when they are being tested for arsenic.

I further made a second experiment to ascertain the influence of free nitric acid. A small quantity of arsenical fluid was put into the Marsh's apparatus, and a mirror was soon obtained. When nitric acid was added, the generation of arsenic hydride seemed to cease, but it began to reappear after about twenty minutes, when no doubt all the nitric acid had been reduced.

Much has been said about the formation of solid arsenic hydride in presence of itric acid, but it is still doubtful whether in presence of zinc and hydrochloric acid this loes not gradually pass into the gaseous state.

In conclusion I wish to remark that although as a rule only a single poison needs to e searched for, many poisons contain two or even more deadly constituents. For nstance, imperial green, chrome-yellow, medicinal tinctures, etc. How often has it not ccurred, the more or less inebriated condition of a person has given his companion a hance of giving him a supposed pick-me-up? I once had to test a stomach for alcohol, nd indeed this body was found, but I also succeeded in isolating weighable quantities of rsenic. It was, therefore, probable this victim had been poisoned whilst in a state of intoxication.

ELECTROLYTIC SEPARATIONS.

By Edgar F. Smith and Lee K. Frankel.

(*Concluded from page* 97.)

A silver dime, weighing 2·4507 grams., carefully cleaned with sodium hydroxide and alcohol, after solution in nitric acid and the expulsion of the excess of the latter, was electrolysed with a current of 0·7 c.c. O-H gas per minute, in the presence of 7 grams. potassium cyanide, and gave 2·1996 grams. silver = 89·79 per cent.

So far as we are aware, this is the first electrolytic method which has been proposed for the separation of these two metals.

SILVER FROM ZINC.

No difficulty is experienced in separating these two metals when in cyanide solution. Our results are as follows:—

	Silver present in grams.	Zinc present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Time in hours.	Silver found.	Difference in per cent.
1	0·1788	100 p. c.	4½	200 c.c.	0·23 c.c.	16	0·1784	− 0·22 per ct.
2	"	"	"	"	0·40 c.c.	"	0·1785	− 0·11 "
3	"	"	"	"	" "	"	0·1779	− 0·50 "
4	"	"	"	"	" "	"	0·1787	− 0·05 "

Applying the same procedure to the separation of

SILVER FROM NICKEL,

the results obtained were equally as good:—

	Silver present in grams.	Nickel present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Time in hours.	Silver found.	Difference in per cent.
1	0·1788	100 p. c.	4½	200 c.c.	0·35 c.c.	16	0·1785	− 0·16 per ct.
2	"	"	"	"	"	"	0·1778	− 0·50 "

SILVER FROM COBALT.

The following are the results obtained :—

	Silver present in grams.	Cobalt present.	KCN in grams.	Total dilution.	Strength of current in c.c. O-H gas per minute.	Time in hours.	Silver found.
1	0·1788	100 p. c.	4½	200 c.c.	0·35 c.c.	16	0·1728
2	„	„	„	„	„	„	0·1747
3	„	„	„	„	„	„	0·1758
4	„	„	„	„	„	„	0·1697

Recalling our experience in separating mercury from cobalt, we reduced the quantity of cyanide to three grams., and obtained :

	Silver present, in grams.	Cobalt present.	KCN in grams.	Total dilution.	Current in c.c. O-H gas per minute.	Time in hours.	Silver found.	Difference in per cent.
5	0·1788	100 p. c.	3	200 c.c.	0·35 c.c.	16	0·1794	+0·33 per ct.
6	„	„	„	„	„	„	0·1782	- 0.33 „

From experiments made to learn the action of the current upon solutions of cobalt in the presence of a great excess of potassium cyanide, we know that the higher cyanide of cobalt—the cobalticyanide—is produced, and may it not be this which in some manner combines with the last traces of mercury and silver to form double cyanides not decomposable by the current strength employed in our experiments, which, if increased, would throw out not only the mercury and silver, but also some cobalt ?

With cadmium, the double compound, if formed, may be more readily broken up, hence the separation is easily made. By reducing the quantity of potassium cyanide with mercury and silver, we afford no opportunity for the production of cobalticyanide in such quantity as to appreciably affect the deposition of the other metals.

COPPER FROM CADMIUM IN THE PRESENCE OF SULPHURIC ACID.

From the fact that it is possible to precipitate cadmium completely from the solution of its sulphate, containing free sulphuric acid (Smith, *American Chemical Journal*, 2, 42), and as copper is also deposited under similar conditions, the separation of these metals, when in this form, would hardly be expected. The experiments given below show that, notwithstanding all this, their separation can be effected under the conditions indicated. The first results were negative :

Ten c.c copper sulphate (0·1975 gram. metallic copper), 10 c.c. cadmium sulphate (0·1828 gram. metallic cadmium), 1 c.c. H_2SO_4 (sp. gr. 1·09), with 150 c.c. water were rolysed with a current generating 0·4 c.c. O-H gas per minute. The copper was precipitated, and with it considerable cadmium.

In a second series of three experiments, similar to that above, excepting that the nt only gave 0·22 c.c. O-H gas per minute, the copper was entirely precipitated, arried down some cadmium with it.

In a third series of three experiments, the sulphuric acid in each dish was ased to 5 c.c. (sp. gr. 1·09). The current gave 0·22 c.c, O-H gas per minute. The r was not completely precipitated, and cadmium had deposited on the copper.

The fourth series was made up as follows :—

(1) 10 c.c. copper sulphate = 0·1975 gram. copper.
10 c.c. cadmium sulphate = 0·1828 gram. cadmium.
10 c.c. H_2SO_4 (sp. gr. 1·09).
100 c.c. water. Current = 0·3 c.c. O-H gas per minute. Time, 12 hours. The copper deposit weighed 0·1968 gram.

(2) Same as (1), copper found = 0·1964 gram.

(3) 10 c c. cadmium sulphate = 0·1828 gram. cadmium, 5 c.c. H_2SO_4 (sp. gr. 1·09). 100 c.c. water. Current as in (1) and (2). Cadmium was not precipitated.

In the fifth series of four experiments, a current generating 0·5 c.c. O-H gas per te was employed. The quantity of acid was increased to 10 c.c. and 15 c.c. cadmium ated together with the copper.

The sixth series included five experiments :—

(1) 10 c.c. copper sulphate = 0·1975 gram. copper.
10 c.c. cadmium sulphate = 0·1828 gram. cadmium.
15 c.c. H_2SO_4 (sp. gr. 1·09.)
100 c.c. water. Current = 2 c.c. O-H gas per minute.
Copper found = 0·1969 gram. copper.

(2) Same as (1), gave 0·1976 gram. copper.

(3) Same as (1) and (2), except that the current generated 0·3 c.c. O-H gas per te. Copper found = 0·1975 gram.

Experiments (4) and (5) were like (3). The copper deposited equalled 0·1969 . and 0·1962 gram. Tabulating the results, we have :—

	Copper required.	Copper found.	Difference in per cent.
1	0·1975	0·1968	– 0·35
2	”	0·1964	– 0·55
3	”	0·1969	– 0·30
4	”	0·1976	+ 0·05
5	”	0·1975	—
6	”	0·1969	– 0·30
7	”	0·1962	– 0·65

The filtrates from the deposited copper were examined for that metal. (7) showed a trace of copper. Cadmium was not detected in the precipitated metal. Holding strictly to the conditions given above, will enable any one to effect the separation of these two metals in the presence of sulphuric acid.

University of Pennsylvania.

ON THE EXPANSION OF FIXED OILS.

By W. T. Wenzell, Ph.M., M.D.*

The relations existing between given volumes of liquids and their temperatures do not appear to follow such simple laws as apply in the case of gases. Whilst it is well known that in general the rate of expansion in every liquid is in direct proportion as the temperature rises, that also liquids expand most rapidly whose boiling points are lowest, and that isomeric compounds such as ethyl fomate and methyl acetate $C_3H_6O_2$, ethyl propionate and methyl butyrate $C_5H_{10}O_2$, etc., having the same boiling point, expand at the same or very nearly at the same ratio, yet, almost every liquid has a co-efficient of expansion different from that of any other liquid, and this co-efficient appears to vary to some extent with the changes of temperature; that is to say, that the rate of expansion is not uniform; a rule which holds good in all volatile liquids.

With the view of ascertaining whether fixed oils are governed by analagous laws, the writer concluded, inasmuch as he could not find data covering these, to investigate the subject. The methods usually followed for determining the cubical expansion of liquids may here be mentioned.

The actual expansion of any liquid may be ascertained by filling with it a small glass bottle whose cubical expansion is known, and weighing the bottle, with the quantity of liquid which fills it, at different temperatures. Another method which is more frequently used, and adopted by Pierre and Kopp in making their numerous determinations, is to observe the apparent expansion of the liquids in a so-called dilatometer, an instrument shaped like a common thermometer, and then correct these observations by taking into account the previously known cubical expansion of the glass.

It would be inaccurate to conclude that the expansion of a liquid between 0° and 100° amounted to one per cent. when the same quantity which filled 100 divisions at the former temperature occupied 101 divisions at the latter temperature, inasmuch as it is impossible to raise the temperature of a liquid without at the same time altering the capacity of the vessel in which it is contained. When a hollow vessel is heated its capacity increases exactly to the same extent as it would do if it was a solid mass of the same material and dimensions. Hence, we must consider the real or absolute expansion of liquids to be the apparent expansion, corrected for the simultaneous expansion of the vessel. These relations may be thus stated:—

The apparent expansion of a liquid is equal to the absolute expansion for the same interval of temperature diminished by the corresponding expansion of the containing vessel, and the co-efficient of absolute expansion in a vessel of any material is equal to the co-efficient of cubical expansion of the material of which the vessel is made. By

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[*] Pharmaceutical Record.

efficient of expansion is understood the increase in volume of the substance ex- l in vulgar or decimal fractions for every degree of temperature. The mean expansion of glass at temperatures of from 0° to 100° C. is $\frac{1}{37634}$ or 0·00002663 for gree; hence these frations represent the co-efficients of expansion of this sub- Therefore, when once the cubical expansion of a vessel is known, the te expansion of any liquid can be easily deduced from its apparent expansion.

ix different samples of fixed oils were used in this investigation, three of which f vegetable origin; the others were derived from the animal kingdom. For a neter a glass flask of 510 c. cm. capacity was used, with a long neck graduated to nth c. cm. at a temperature of 62° F. The flask was filled with the oil to O, ated with the stem immersed in a double water bath, care being taken that the bath and oils were exactly at 62° F., a temperature easily obtained in San sco without artificial aids. The whole apparatus was covered with felt and n envelopes, and every care taken to insure an even temperature. Through the the felt covering holes were cut to allow the stem of the flask and thermometer aised for inspection.

he heat was applied by a diminutive coal oil lamp, so regulated that an expansion oil not to exceed 1 c. cm. per hour was insured.

uring the heating of the oils the flask was moved up and down about every five es, in order to heat the water surrounding it uniformly.

he following were the results obtained:—

Olive Oil.

Dilatometer.	Thermometer.	Difference.	Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—	506 c. cm.	92° F.	5°
501 ,,	67° ,,	5°	507 ,,	97° ,,	5°
502 ,,	72° ,,	5°	508 ,,	102° ,,	5°
503 ,,	77° ,,	5°	509 ,,	107° ,,	5°
504 ,,	82° ,,	5°	510 ,,	112° ,,	5°
505 ,,	87° ,,	5°			

Mustard Seed Oil.

Dilatometer.	Thermometer.	Difference.	Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—	506·1 c. cm.	98°·5 F.	5°
501 ,,	67° ,,	5°	506·6 ,,	95° ,,	5°
502·1 ,,	72°·5 ,,	5°	506·8 ,,	96° ,,	5°
503·4 ,,	79° ,,	5°	508 ,,	102° ,,	5°
505·1 ,,	87°·5 ,,	5°	509 ,,	107° ,,	5°
505·7 ,,	90°·5 ,,	5°	510 ,,	112° ,,	5°

It will be seen that, although the above figures do not all give whole numbers, yet xpansion of mustard seed oil agrees with the olive oil, the expansion of each re- vely being 1 c. cm. for every 50° F.: 72·5°—62° ÷ 2·1 c. cm. = 5.

Cotton Seed Oil.

Dilatometer.	Thermometer.	Differerence.	Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—	506 c. cm.	92° F.	10°
502 ,,	72° ,,	10°	508 ,,	102° ,,	10°
504 ,,	82° ,,	10°	510 ,,	112° ,,	10°

Lard Oil.

Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—
510 ,,	112° ,,	50°

Castor Oil.

Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—
510 ,,	112° ,,	50°

Sperm Oil.

Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—
510 ,,	113° ,,	50°

Cod Liver Oil.

Dilatometer.	Thermometer.	Difference.
500 c. cm.	62° F.	—
510 ,,	112° ,,	50°

These experiments show that 500 c. cm. of either of these oils require 50° F. to expand 10 c. cm. or 5° F. to expand 1 c. cm. As the co-efficient of expansion is expressed in Centigrade degrees, therefore a reduction from Fahrenheit to Centigrade is necessary.

$$50° - 32° \div 9 \times 5 = 10° \text{ C.}$$

Ten degrees C. increase 500 c. cm. of oil to 510 c. cm., which equals 1° C. to each c. cm. in the 500 c. cm., hence $\frac{1}{500} = {\cdot}0020000$ the apparent rate of expansion, and co-
efficient of apparent expansion, ·00200000
+ Co-efficient of expansion of glass, ·00002663
= Co-efficient of absolute cubical expansion of oils, 0·0202663

In conclusion, the author would direct attention to facts elicited by this paper, that, unlike all volatile oils and liquids, the fixed oils made the subject of this paper have the same co-efficient of expansion, and that their rate of expansion is uniform for all degrees of heat from 62° F. (16·6° C.) to 112° F. (44·4° C.)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Detection of Yolk of Egg in Foods. Wichelhaus (*Pharm. Zeit.*, 1890).—Although phosphoric acid or its salts are insoluble in ether, this fluid is an excellent solvent for lecithin, a product of decomposition of vitellin, the albuminous body of the yolk. This lecithin, when saponified, yields a notable quantity of phosphoric acid, which may be estimated in the ordinary way after ignition of the soap. As the theoretical yield is 9·2 per cent. phosphoric anhydride the proportion of lecithin may be calculated by multiplying the phosphoric acid formed by 10·87. If not found in too minute quantity, an analyst may safely conclude the food contained yolk of egg.

L. de K.

Analysis of Sodium Aluminate. G. Lunge (*Zeitschr. f. Angew Chemie*, No. 8, 1890).—This compound is used in the manufacture of soap, and is an ingenious device for introducing excess of mineral matter (alumina). The analysis presents, of course, no difficulty when performed in the ordinary gravimetric way, which is, in fact, the only an when there is any notable excess of silica. If, however, there is no danger of ing the case, the author proposes the following volumetric process, which is ntly accurate for technical purposes.

bout 20 grams. of the compound are dissolved in a litre of water; 10 c.c. are mixed henol-phthalein and titrated whilst hot with normal hydrochloric acid. This gives a. Methylorange is now added and more acid slowly added until the fluid has permanently reddish. This will give the alumina. It is as well to repeat the n, adding the bulk of the acid at once, and then drop by drop. L. DE K.

STIMATION OF MINERAL OILS IN FATTY OILS. GRITTNER (*Zeitschr. f. Angew*, No. 9, 1890).—One of the most convenient processes is the one devised by (see ANALYST, 1888, p. 184), which consists in saponifying the sample with c soda, and extracting the dry mass with chloroform. The author finds, r, that when the mineral oil preponderates the extraction becomes tedious, and refore proceeds as follows:—About 3 grams. of the sample are saponified in a in dish with 20 c.c. of a solution of 25 grams. of caustic soda in a litre of spirits e. After the alcohol has evaporated the mass is mixed with sand and then put cartridge which is covered with a little filter. The extraction by means of orm is performed in the Soxhlet as usual. The chloroform is finally distilled off residual oil dried at 100° C.

the chloroform is not pure it is best to redistil it over sulphuric acid. The sand ust be well washed with hydrochloric acid to free it from lime, otherwise there er of forming a lime soap, which is far from insoluble in chloroform. L. DE K.

EW TEST FOR NITROGEN. E. DONATH (*Chem. Zeit.*, 1890).—About ·05 gram. substance is boiled with a solution of 1 gram. potassic permanganate and 20 c.c. rated potash ley, adding more permanganate if required. After dilution with the excess of the permanganate is destroyed by adding a little rectified spirit, and uid filtered off from the precipitated manganic hydrate. As part of the nitrogen n oxidised to nitrous acid, it is easy to get the test for this acid by the addition rochloric acid, potassic iodide, and starch. Sometimes the oxidation proceeds and a nitrate is formed, which may be shown by the brucine test.

s the author succeeded in getting nitrous or nitric acid from pepsine, urea, , coal, etc., he believes it to be a good general test.

his reaction will also explain why in the albumenoid ammonia process only a of the nitrogen is converted into ammonia. L. DE K.

OLUMETRIC ESTIMATION OF CADMIUM IN PRESENCE OF ZINC. W. MINOR (*Chem.* o. 21.)—The sample is dissolved in hydrochloric acid and filtered off from any Besides cadmium and zinc, the liquid contains iron, and often traces of arsenic. dmium is now precipitated with hydrogen sulphide, and the cadmium sulphide d and weighed. It, however, always contains more or less zinc, often as much as ent. It is, therefore, redissolved in hydrochloric acid, and after boiling, treated odium hydrate in excess, which throws down the cadmium and redissolves the zinc. The amount of the latter (reckoned as sulphide) is then ascertained by titrating with standard solution of sodium sulphide, and deducted from the joint weight of cadmium and zinc sulphides.

An alternate process, suitable under some conditions, is based on the following principle: Neutral cadmium solutions are precipitated by sodium hydrate according to the equation

$$Cd\,Cl_2 + 2\,Na\,HO = Cd\,H_2O_2 + 2\,Na\,Cl.$$

Therefore 1 c.c. normal soda = ·0558 cadmium.

In applying this process, the solution must, of course, be free from excess of acid and any heavy metal. Therefore, in presence of zinc and iron, the last is first precipitated with ammonia, and the filtrate after being neutralised with hydrochloric acid, treated with caustic soda. After washing the cadmium hydrate, it must be dissolved in hydrochloric acid, and the excess of acid completely expelled by evaporation. The residue is then fit for titrating with soda. As indicator the author uses either red litmus paper until faint alkalinity is observed, or paper moistened with sodium sulphide until no more cadmium reaction is observed. The test analyses are satisfactory. L. de K.

Estimation of Tellurium in Minerals. E. Donath (*Zeitschr. f. angew Chemie*, No. 7, 1890).—The usual plan is to throw down the metalloid with sulphurous acid, although the author preferred the fluid obtained by acting with zinc on a mixture of sodium bisulphite and hydrochloric acid. This process not working well in presence of bismuth and other metals of the fifth group, the author now prefers to reduce with pure glucose (free from calcium compounds).

About 4 grammes of the finely powdered ore are treated in a porcelain dish, with the usual precautions with small quantities of nitric acid. The excess of acid is then evaporated off, without, however, decomposing the nitrates of iron, bismuth, or copper, as otherwise the tellurium would pass into the *ic* state. After rubbing the dried mass with an agate pestle it is moistened with strong sodium hydrate, which will cause the mixture to get hot, whilst black copper oxide separates. After about half an hour some more solution of soda is added, the liquid is filtered off and now boiled for twenty minutes with glucose, which throws down the tellurium. This may now be collected on a weighed filter, or made into oxide ($Te\,O_2$) as follows:—

The precipitate is rinsed off from the filter with a warm mixture of two volumes of nitric acid and one volume of water, containing also a few drops of sulphuric acid. The solution is evaporated and the residue ignited and weighed. The process is easy and sufficiently accurate for technical purposes. L. de K.

REVIEWS, NOTES, Etc.

A Handbook of Quantitative Analysis, by John Mills, of the Normal School of Science, and Barker North, Associate of the Normal School of Science, Illustrated.—London: Chapman and Hall, Limited, 1889.

This is a very well-written and complete little book for students preparing for any examination in ordinary quantitative analysis, and it is claimed to be sufficient for the Associateship of the Institute. Whether this be so or not, it forms a series of well-

ed exercises for students commencing the subject. It would be somewhat in the
of "breaking a butterfly" to indulge in a criticsm from the point of view of a
al analytical chemist, otherwise we could indicate one or two points where the
s fall short of what would be the true practice to ensure absolute accuracy, and we
absolutely agree with the short summary of the conclusions to be drawn from the
of a water analysis. Still, on the whole, taking the book for what it is intended,
useful compilation, and given a practical teacher to point out where it is to be
ed and occasionally modified, it will be found very convenient for the use of
ts.

ETECTION OF GROUND NUT-SHELLS OR OLIVE STONES IN PEPPER.—In reply to a
ondent we give the following method which is employed in the Paris Municipal
tory by M. Girard :—

) *Preparation of the reagent.* This is a solution of dimethylparaphenylene-
e and sodium bisulphite and is made as follows :—10 grams. of the ordinary com-
l dimethylaniline are mixed in a basin with 20 grams. of pure concentrated hydro-
acid. To this mixture 100 grams. of crushed ice are added and 100 c.c. of a 7
t. solution of sodium nitrate is cautiously introduced, a little at a time with
t shaking, and the whole is set aside for half-an-hour for the reaction to become
ly complete. 30 to 40 c.c. of hydrochloric acid are then introduced, together with
ms. of pure tin foil, and the reducing action so caused is allowed to continue for
r, at the expiration of which sufficient granulated zinc is introduced to precipitate
. The liquid is then filtered and neutralised with solution of potassium carbonate,
is added until a permanent cloud forms, and acetic acid is carefully dropped in
he turbidity just appears. 10 grams. of concentrated solution of sodium bisul-
are then added, and the whole is finally made up to two litres with distilled

2) *Application of the reagent.* 2 c.c. of the reagent are placed into a porcelain
and a good large pinch of the pepper is sprinkled into the liquid and allowed to
for some minutes. Pure pepper either remains without change or is only tinted
pale rose, while all the adulterating particles of olive stones or other sclerogenous
are coloured a bright carmine. If some water be now added the heavier red par-
collect together at the bottom, and a fairly good quantative idea can thus be
ed.

he indication thus observed may be farther confirmed by repeating the experiment
solution of sulphate of thalline (·5 gram. in 100 c.c.), which colours the sclero-
s tissue bright orange.

REPARED COCOA.—We have been requested by a correspondent to give some par-
rs on this subject, and we now intend to do so, although, of course, all the matter
ly perfectly well known to the professional section of our readers, who are, there-
warned to skip this paragraph, and not to grumble at our occasionally providing a
reading interesting to the general public. Cocoa is, in its natural state, highly
lating, but at the same time a very rich and somewhat indigestible substance, con-

taining a large amount of a fat known as cacao butter. To prepare cocoa for table, it is necessary that the amount of this fat should be reduced. This is attained by using one of two methods, viz.: (1) The roasted and ground cocoa is mixed with starch and sugar so as to dilute its richness; (2) before grinding the cocoa, it is heated and pressed, whereby a considerable amount (generally nearly one-half) of its natural fat is squeezed out. This latter method is decidedly the more desirable one, because not only is the fat removed, but all the other nourishing and stimulating principles are at the same time concentrated. A typical cocoa of English manufacture by this process is found in the well-known Cadbury's cocoa essence. This article has always been, in our experience, exceedingly constant in its nature, and the last time we analysed it we found:

Fat		28·12
Theobromine		·95
Natural starch, sugar, fibre, etc.		40·85
Soluble albumenoids, 6·61 Insoluble albumenoids, 14·73	Total flesh formers	21·34
Natural mineral matter		4·94
Moisture		3·80
		100·00

Here then is what constitutes, in our opinion, a specimen of a properly-prepared cocoa, nothing added to dilute its flesh-forming or stimulating properties, and nothing deducted except the indigestible fat. There are, of course, other excellent prepared cocoas in the market, but we have purposely chosen Cadbury's as the type to illustrate these remarks because it is really the standard English article, and was, we believe, the first so manufactured. Indeed, until it was brought out, the only style of cocoas known to English consumers were compounds that produced, when cooked, what was practically little more than sweetened starch flavoured with real cocoa. Even now persons are to be found who prefer such articles, or who simply drink what their grocers send them and call it cocoa. If our correspondent happens to be one of these, let him try the real article, prepared by the fat extraction method, and give up buying extra starch and sugar under the name of prepared cocoa. To give a still more striking confirmation of our remarks let our readers glance at the following average amounts of flesh-forming ingredients present in the substances named:—

Natural cocoa	13·00
Good wheaten flour	14·00
Cocoa prepared with added starch and sugar	6·00
Cocoa prepared by extraction of fat (Cadbury's)	21·00

Exalgin may be distinguished by the following simple test from acetanilid and phenacetin: 1 gram. is dissolved in 2 c.c. chloroform (acetanilid requires 6 c.c. and phenacetin 20 c.c. chloroform) and 20 c.c. petroleum ether (sp. gr. 0·650) added; the solution should remain clear. 10 per cent. phenacetin and 20 per cent. acetanilid can be detected by the formation of a precipitate after standing a short time.—(E. Hirschsohn).

A Test for Hydrogen Dioxide.—The solution to be tested is made alkaline, and then a soluble neutral salt of lead or copper added; a deep brown-red precipitate, rapidly changing to red, and finally to white, indicates hydrogen peroxide. In concentrated solutions effervescence is also to be observed. Ozone solution (Lender's) does not give *this test.*—(A. O. Gawalowski.)—*Am. Journ. Pharm.*

THE ANALYST.

JULY, 1890.

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Owing to the pressure of the Society's matter this issue of the ANALYST *is entirely devoted to the same.*

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

AN ordinary meeting was held at Burlington House on Wednesday, the 11th ult., the President, Mr. Adams, in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected as members:—S. J. Steel, F.C.S., Blackheath, as Associate; T. H. Pearmain, Assistant to Mr. Allen.

The following gentlemen were proposed for election as members:—G. A. Milne, F.C.S., Analytical Chemist, Norton Malton; H. Barclay, Analytical Chemist, Workington; Edgar Richards, of the American Inland Revenue Department.

The following papers were read and discussed:—

"On Dyed Sugar."—By C. E. CASSAL.

"On some Recent Advances in our Knowledge of the Sugar Group."—By Dr. W. J. SYKES.

"On some Points in the Analysis of Milk."—By H. DROOP RICHMOND.

"Notes on the Chemistry and Detection of certain Hop Substitutes."—By A. H. ALLEN.

The country meeting of the Society will be held at Gloucester on the 25th inst. Full particulars will be duly announced.

REMARKS ON THE BITTER OF HOPS AS DISTINGUISHED FROM THE BITTER OF QUASSIA AND OTHER "HOP SUBSTITUTES."

BY MATHEW A. ADAMS.

Read at Meeting, May, 1890.

IT was at the commencement of 1875 that a consideration of the bitter principle of the hop was first brought under my notice with a view to distinguish it by chemical or physical means from the bitter of quassia or other substitutes of a similar nature, reputed to be used in the composition of sundry proprietary articles variously called "Hop Substitutes," "Hop Supplements," "Bavarian Hop Bitter," and so on, which articles were extensively advertised, and said to be largely used to replace the true hop bitter in the brewing of beer.

The "Hop Growers' Association," as it was called, applied to me for advice and assistance in the endeavour to discover some means for distinguishing, by chemical analysis or otherwise, between these substances. Positively nothing appeared then to be known concerning these bitters, and being unable myself to supply any information, upon my advice, certain prominent chemists who had made the question of food analysis a special study were consulted, but without any satisfactory result.

In September, 1875, the Association offered a reward by advertisement in various professional papers of £100 for the discovery of a method for detecting the presence of quassia or other substitutes for hops in malt liquor. This offer, if I remember right, was to hold good for twelve months. Two others and myself were appointed to test and adjudicate upon the methods of any claimants for the reward, but no claim was made and not a scrap of information obtained. So the offer of a reward lapsed, and the whole proceeding was reckoned a failure.

Meantime I had been turning the thing over in my mind, and was invited to undertake the investigation myself, and a small sum was set apart as remuneration to me for doing so.

I was supplied with samples of the various hop substitutes then in the market. I obtained samples of various medicinal tonic bitters which appeared to enter into the composition of the hop substitutes, and worked at the thing myself with the result that, early in 1877 I was able to write to the Chairman of the Hop Growers' Association announcing my ability to detect foreign bitters in beer, and offering to subject myself to a trial. Twenty-seven samples were sent me to test my ability to do so, some of them had been specially prepared by the committee, and my analysis showed that nine had been bittered with foreign bitters and eighteen were not so bittered, and this was correct. The eighteen pure samples were mostly of porter from various parts of London. The Association were very much disappointed that quassia had not been discovered in any of these London beers, and they then proposed to undertake a roving expedition throughout England to find a beer with foreign bitter in it. I had no objection to that, but could not see the fun of doing all this work without being paid for it, and offered to disclose the secret of my process for the lapsed reward, or to do any number of analyses at a fixed fee. This they did not consent to, but put themselves into communication with some other chemist, but who that chemist was I never enquired and never knew. That was the end of my dealings with the Hop Growers' Association, and the subject of hop bitters, so far as I was concerned, was put altogether on one side, and no one, I imagine, will blame me for not being in a temper to publicly disclose the results of my researches for the benefit of a rival by whom I had been supplanted. In 1887, as you all know, Mr. Allen invited communication on this subject from members of this Society, and at the May meeting, which was well attended and wholly given up to a consideration of the matter, Mr. Allen read a very interesting paper on the subject, and many others, either by letters or voice, also contributed to the discussion that followed. The part that I took was to testify to the practicability of distinguishing between the hop and hop substitutes, which I did by private comnication with Mr. Allen before he read his paper, and also subsequently at the blic meeting. In February, 1888, Mr. Allen again brought the subject forward discussion, and I explained my method, so far as the use of sulphuric acid is cerned, and subsequent to this in a foot-note on page 46 of the ANALYST, Mr. Allen ports that he had succeeded with the sulphuric acid treatment on the sample of hops, ich gave the sensible bitter extract (by the ordinary method of applying the lead t), and found all trace of bitter had disappeared, even when the concentrated ution was agitated with chloroform and the evaporated chloroform solution tasted.

It is not that I am over concerned to identify myself as the original investitor in the field that I have ventured to occupy a few minutes with this historical tement. My object is to repel an insinuation that I had unaccountably kept secret knowledge on this subject. For the credit of our Society I take leave to say at there is no more liberal-minded body of men to be found than the members this Society. I have never known an instance where information has been withld for private interest or refused by one member to another when applied for. e value of this freemasonry among us is incalculable; but for this the public analysts Great Britain could not have been the competent men they now are. Aye, nor uld the present knowledge of food analysis be what it now is; and in passing I ould like to say it has often occurred to me that the strongest argument to be und against the fusion of this Society with any other is the possible risk that to me extent the opportunity for this freedom of interchange of thought and exrience within the limits of our special field might be jeopardised. Since the February eeting in 1888, the subject again fell into abeyance, and it was only a week or two o that Mr. Robert Norton, the member for the Tonbridge division of Kent, called on me to ask whether I could furnish any information on the chemical aspect of e question for the Select Commission of the House of Commons now enquiring to the causes of the depression in the hop industry. I consented to do so, and d give evidence on May 1st, and now ask your kind attention to a few hastily put tother remarks on the subject.

Anyone by the unaided sense of taste may observe a wide difference between rious bitters in the duration of their effect upon the palate, and this is a characteristic at seems to divide bitter substances, sharply, into two categories:—

1.—Fixed Bitters.

2.—Fugitive Bitters.

hese characteristics manifest themselves

1.—Physiologically in their effect upon the body, and also—

2.—Chemically in their behaviour under chemical treatment.

A fixed bitter affects the organ of taste in a way that is enduring, and not only es the effect of the bitter last a long time, but also for the time being it appears to erwhelm the taste organs; so that these taste organs require a considerable time to cover their entire freedom to repeat their office. On the other hand a fugitive bitter s not anything like so penetrating an effect, and the effect lasts but a comparatively ort time, leaving the palate clean and free to appreciate other flavours. Moreover, e can, as it were, taste through the bitter of the fugitive bitter, whereas the fixed bitter covers everything, and, so to speak, monopolises the sense of taste, hangs about the mouth, and perverts other flavours for a long time.

In short, the stimulus to the gustatory fibres of the glossopharyngeal nerve, is in the one case transitory, but in the other long continuing, for which we shall, I think, presently find an explanation. The hop bitter will serve as an example of what I call a fugitive bitter and quassia of a fixed bitter. It is not only from a physiological, but in some respects also from a chemical point of view, that this characteristic distinction is displayed.

Quassia and its allies will stand a deal more rough usage, so to speak, and yet survive, whilst the hop bitter is destroyed; in a word the one is a much more stable compound than the other, but I take the liberty of applying the terms "fixed" and "fugitive" in a chemical sense, simply because by certain methods of chemical treatment, presently to be described, the fugitive is driven away whilst the fixed is left behind.

The proximate nature of the hop bitter is ascribed to several resins varying in intensity of bitter, but I hardly think its true nature has yet been positively and definitely made out. I suspect that a part, if not the whole, of this bitter is of the nature of a glucoside; at all events, this view fits in very well with the facts. For instance, fermentation removes a large part of the bitter, and *boiling a 2½ per cent. decoction of hop with 2½ per cent. of sulphuric acid under a reflux condenser for two or three hours every trace of bitter is entirely removed.* But quassia and its allies are not at all affected by this treatment. This is the key note of the distinction between the fixed and fugitive bitters—the latter being more or less of the nature of the glucosides, on this account disappearing by conversion into glucose in the acidified decoction, and in the mouth by digestion through the agency of the ferment proper to the saliva—and so we can now understand how it is that the palate when dealing with a fugitive bitter clears up so quickly, while, on the other hand, a fixed bitter, being insusceptible of fermentive change maintains its bitter effect so long.

As regards the argument that the fugitive character of the taste of hop bitter is due to its glucoside nature, allow me to draw your attention to the fact that salicin, a very bitter substance, but also a well-known glucoside, has this same fugitive characteristic in respect of taste, for when a saturated solution is placed on the tongue all bitterness soon vanishes.

The hop bitter, however, though it is of the nature of a glucoside, is by no means easy of conversion, so that prolonged boiling with the sulphuric acid is necessary, and fearing that this somewhat severe treatment might possibly, to a certain extent, attack some of the fixed bitters, it seems as well to take advantage of the selective power of the basic acetate of lead to assist and hasten the removal of the bitter, so that the process I employ is as follows:—

First Process.

The decoction is made to boil, and basic acid of lead solution is added until the point of saturation is just reached. The boiling is continued for some time, after which the precipitate is filtered off, and the filtrate at once treated with sulphuric acid until there is a decided excess. The lead sulphate is filtered off, and the clear acid filtrate gently heated until by evaporation it is reduced to a small bulk. Chalk is then added to remove the acid, the decoction filtered, and if nothing but hop has been used in the decoction, all bitterness will have disappeared.

But if quassia or any of the hop substitutes has been used it will be as bitter as ever, and in proportion as it is concentrated more bitter; if it was a mixture of hop and quassia, the hop bitter will have been removed, leaving the quassia behind. I have spoken of quassia as an example, but the same holds good for Gentian, Chiretta Calumba, and a very large number of vegetable bitters, probably all of the fixed or unfermentable bitters, and certainly for all the various so-called hop substitutes that have come to my hands. There is, of course, no difficulty in applying these principles to the detection of the false bittering of beer, and to the best of my belief so long as we have to deal with new hops the process as described is always reliable.

I have said that the hop bitter is not easy of conversion; in some cases it is more stubborn than others. My original experiments were conducted upon samples of new hops only, but recently, a few days ago, I was experimenting upon some old hops—too old for use in brewing—and I was surprised to find that there was a certain amount of residual bitter left in the final filtrate at the end of the experiment, so the process was repeated with additional care, but the second result was, perhaps, more bitter than the first. Thinking that it was possible some of the precipitate might have been redissolved by excess of lead acetate, in a third experiment the lead precipitation was done in fractions, care being taken in the first and second precipitations to keep well within the point of saturation, and so prevent, as far as possible, re-solution by the lead acetate; and yet the result was bitter, even more bitter than in either of the two first experiments. From this it appeared that the more carefully we precipitated the more completely we laid bare residual bitter. A similar result occurring when we treated other samples of *old hops* there seemed little doubt but that the *age* of the hops had something to do with causing the difficulty. For the moment we were nonplussed, but by the following expedient the difficulty was overcome in a very satisfactory way.

SECOND PROCESS.

Starting with the decoction of this obstinate sample, we raised it to the boil, and added barium hydrate solution till an alkaline reaction was obtained. This threw down a rich chocolate brown precipitate, which was filtered off, and the bright, rosy red filtrate was made just acid with sulphuric acid heated and then filtered, and the filtrate evaporated to about a quarter of its original bulk; by this time most of the bitter was gone. Then the lead treatment was proceeded with as in the first process, and the result was quite satisfactory, and for all practical purposes bitterless; and so far as my experience with the process has gone, it has never failed to remove hop bitter even in the most obstinate of old hops that I have yet come across; at the same time it has no effect upon quassia.

So that by this improved process it would appear to be possible not only to determine the presence of a foreign bitter, but even to discriminate between a beer that has been bittered by old from another that has been bittered by new hops.

I cannot pretend to have given this new process an extensive trial. I have not had the time to do so, but seeing it has proved successful in cases where the first process had failed, only by reason of the age of the hops used, I am greatly in hopes that it will be found effectual in all cases, but I need hardly say that it is my intention, as time permits, to apply the process to every variety of hop, both as regards age and source, and in the improbable event of failure on account of some undiscovered peculiarity in respect of a particular sample of hops due to age, source, or other reason, we can with confidence fall back upon the simple, if somewhat severe, method of boiling with sulphuric acid, so that in any case the difficulty is solved.

So much for the chemical aspect of the case. Let us pass to a consideration of the fixed and fugitive bitters from a health point of view. This, although essentially a medical question, can be treated in a popular common-sense way, and if we continue to take the hop and quassia as illustrative examples, it appears to me that there is sound common sense reason for making a clear distinction between the two in respect of their general effects upon the human body. Very little precise knowledge is available as to what becomes of quassia when taken into the system, but from what we know of its behaviour under chemical and fermentive treatment, we may be quite sure it is not easily changed, and in all probability, like quinine and strychnine suffers little or no decomposition in the body, but is excreted with the urine, and probably also with the fœces in an unaltered condition ; that it is carried along the whole tract of the intestinal canal, a point of considerable importance, there can be no doubt; sufficient evidence of this is found in the effectual vermifuge action it exerts upon ascarides (worms that reside in the lowest part of the bowel), so that with quassia we have to do with a drug that is incapable of digestion, and in this respect one that behaves like quinine and strychnine, drugs that have a more profound effect upon the body than almost any with which we are acquainted. On the other hand there is little or nothing to show that the hop bitter can exist for any considerable length of time in the body without undergoing complete digestion, and being assimilated into the system and turned to account as food. Well, you may be disposed to say all this may be true of the ultimate results of these substances when introduced into the body, but is it not also true that both manifest tonic properties, improving the digestion and so forth? Undoubtedly that is so, but even in this respect it is obvious that their action is not identical. The hop exerts a tonic action that is temporary lasting only so long as it remains undigested, for as soon as by digestion it has been converted into food it ceases to be a tonic. On the other hand, the indigestible bitter of quassia from first to last keeps up its tonic stimulation. No doubt in certain morbid conditions of the body this may prove an advantage, but in the normal state of health the digestion does not require to be perpetually goaded to action.

The word tonic, as we are now employing it, implies a special kind of stimulus upon the functional activity of the alimentary tract, and we may be quite sure that intervals of non-stimulation, in other words rest, are just as necessary for that part as any other part of our anatomy. It may be all very proper and salutary to rouse the energy of appetite and digestion, but in health to maintain an unnatural stimulation, and so cause a continuous nervous tension in the parts affected, it appears to me, must be prejudicial; it is as though a bow were always kept strung. I have often pictured to myself the beneficial effect of a transient tonic such as we have in the fugitive bitter of *the hop*, by the parallel effect we observe after a plunge into the sea and out again;

reaction follows upon the sudden short stimulus, and the benefit is obvious. On the other hand, if the immersion be prolonged the bather is not benefited; exhaustion takes the place of reaction, the bow has been too long strung. It is wonderful what tolerant bodies we inhabit, but we may depend upon it the tendency of unnecessary and prolonged stimulation of the digestive organs by the habitual use of fixed bitters is more or less prejudicial.

Turning to another point, I can fancy some reluctant convert to these views might say, "Ah! but how about the soporific, drowsy effect of the hops? Is not that a set-off, or even more than a set-off, for the injury you impute to the fixed bitter?" Well, let us see. In the first place, upon what authority does the statement rest that hops have a soporific effect. I have looked through all the books on *materia medica* that are at this moment accessible to me, and I can find very little justification for the imputation that the hop is soporific at all; the fact is, from a therapeutical point of view, the hop occupies a very humble place, so much so that by some modern writers, as Ringer for instance, is not even mentioned. This is what Garrod says:—"Hops are tonic and stomachic and slightly narcotic; in the form of bitter beer, taken with meals, they form a useful aid to digestion in some cases of atonicdyspepsia. The volatile oil is probably the narcotic principle, and in the form of a pillow, hops have been found anodyne and narcotic."

And Negilgan says:—"Much difference of opinion exists as to the therapeutical properties of hops; they are generally stated to be narcotic, but from the experiments made with them on animals by Majendie and others it would appear that this effect is not manifest when they are given internally, no matter how large the dose."

And Periera says:—"Tho odorous emanations (vapour of the volatile oil) of hops possess narcotic properties; hence a pillow of these cones promotes sleep, as I have several times witnessed." "Moreover, we are told that stupor has occasionally been induced in persons who have remained for a considerable time in hop warehouses. The lupulinic grains are aromatic and tonic. They appear also to possess soothing, tranquilizing, and, in a slight degree, sedative and soporific properties. But the existence of any narcotic quality has been strongly denied by Dr. Bigsby, Majendie, and others." "I have tried it at different times," says Majendie, "both the lupuline (lupulinic grains) in substance and its different preparations, on animals, but I have never observed that it is a narcotic, although this property is one which is most strikingly displayed in experiments on animals."

As regards the reputation of the hop pillow, which has been handed down from book to book, it is very doubtful if there be much in it—for although I live in the midst of the chief hop-growing districts I never knew of its actually being put in practice—I quite believe that it is an old legend that gained more credence than it deserved owing to the anecdote that King George III., being a sufferer from faceache, derived comfort and got sleep by the use of a hop pillow that a loyal subject of Kent presented to him. Be this as it may, so far as medical experience goes, there is really nothing to countenance the idea that the hop, except in its volatile constituents, contains anything soporific, and in point of fact I do not believe the hop contributes to the finished beer, so far as the consumer is concerned, anything beyond a delicious aroma and an agreeable salutary bitter.

I can see nothing but fallacy in the argument that "*stock beer*" is more heady and intoxicating than a "*present use beer*," because the former contains a pound or two more hops per barrel. Depend upon it, the difference has nothing to do with the hops; they, as we all know, are introduced by the brewer to give the stock ale its keeping quality, and it is the keeping, the maturing, that gives the beer its "*drowsy influence.*" It is very well known that the ageing of beer is attended and accomplished by further fermentative and other changes which take place in the cask, and the products of this cask fermentation, which are of a totally different character to those of the primary fermentation, the alcohols, æthers, etc., that are produced, although but little further attenuation takes place, immensely increase the intoxicating property of the beer. No doubt they are amylic or other heavy alcohols; indeed, practical brewers' chemists acknowledge this. One, for instance, says: "It is useless to disguise the fact that English beer contains fusel oil. If not, I ask why it is that lager beer is not intoxicating; it frequently contains more alcohol than mild ale;" and again he says, "English beer undoubtedly contains heavy alcohols, æthers, and fatty acids, to which it owes the properties (intoxicating) wanting in German beer."

On the other hand, we have not far to seek for the explanation of the immense improvement that has taken place as respects the less intoxicating effect of modern as compared with old-fashioned beer. From beginning to end, science has fairly revolutionised brewing. There is no need for me to remind you gentlemen of the intelligent control that is now maintained over the temperatures employed in the mashing, in the fermentation and attemperation, nor of the importance of the skilful care that is now bestowed upon the selection and proportioning of the malt and malt adjuncts, and the microscopic watchfulness over the yeast. These facts considered, it is no marvel that beer has improved; it is far more of a marvel that the brewers' chemists, to whom the whole of the credit is really due should be ready with self-denying modesty to conceal their own just merits in an unfair attempt to fasten upon the indispensable, unoffending hop the demerits of the old-fashioned beer.

Gentlemen, I hope I have not detained you too long. If you think so, I must plead as excuse what took place when I was before the Committee of the House of Commons on the hop question a few days ago. I was asked, "Should you have any objection to lay before the authorities at Somerset House the full results of all your experiments?" to which I replied, "I should, as a matter of course, occupying the position I do hold of President of the Society of Public Analysts, feel the greatest pleasure in communicating all I know upon this subject to the Council of the Society of Public Analysts, and it would come, as a matter of course to be general knowledge. I have no wish or desire to keep the matter secret."

Question.—"And assuming that it might be necessary to make still further experiments, do you think we might rely upon the help of the body of Public Analysts over whom you have the honour to preside? Do you think they would help you in carrying out further experiments?"

To which I replied: "I think the history of our society would show that all that has been done to advance the methods of analysis of food has almost entirely originated with our society, and there has never been, so far as I am acquainted with the matter, the smallest indication of reticence on their part, in withholding anything like a new method, or new knowledge, which has come to the knowledge of the members individually. The utmost liberality has been shown in that respect; in fact I think we have not had justice done to us."

Gentlemen, this contribution is a first instalment towards the redemption of that pledge. We all know that for an enquiry of this kind, besides skill, time and patience are needed to bring it to a perfect issue, and we well know that all that is required to secure the needful patience and skill is to convince you that occasion has arisen that will justify us in the expenditure of the time necessary for the investigation, and I feel sure, in the name of the society, I may venture to say that if legislation is dependent upon a satisfactory test for the detection of bitters other than hops in beer, we may be trusted to supply both men and methods capable of a complete solution of the problem.

Discussion.

Dr. Sykes (who had temporarily taken the chair) said they would all agree with him that they had heard a very interesting paper, both from a physiological and chemical point of view, and he invited discussion.

Mr. Gordon Salamon said: They must all join in the opinion that the President had communicated a most interesting paper, one which he could not help thinking embodied the result of his cogitation over the cross-examination to which he was recently subjected before the Select Committee of the House of Commons. For poetic treatment the paper left nothing to be desired, but from a scientific standpoint he was bound to take grave exception to much that had been said.

Before discussing it from that point of view, he would, however, venture to suggest that the President had chosen a most inopportune moment for reading his paper. He disclaimed all personal feeling in stating his opinion that the President should have brought his process to the notice of the Society instead of giving it publicity for the first time to a Select Committee of the House of Commons; more especially as the Committee might be contemplating a recommendation of legislation upon the strength of that process, backed as it was by the President's position in respect of their Society. They had been told that it was only a fortnight since that he received an intimation from Mr. Norton that he would be wanted to give evidence before the Committee, but he could not think that such a statement relieved the President from the duty of first of all making the Society's journal the repository of his process. The President had developed his process fifteen years ago, but it was only now, when he had given his evidence in the name of the Society, and the inquiry relating to the science of the subject had closed, that he came to ask them for an opinion as to the merits of his process. He thought that the course taken was much to be deplored, because it was certain that if he had first of all submitted the question to them, the cross-examination would have been anticipated, and the President would never have given evidence which had resulted in unintentionally misleading the Committee. The President was questioned by Sir Henry Roscoe with a facility and ability which they all knew him to possess, with the result that the President had come there that evening to inform his brother analysts that he had now abandoned his process—one, be it remembered, which he had informed the Select Committee only a fortnight previously, would enable him to go into the witness-box and swear to the presence of hop substitutes, and would, moreover, permit of the enforcement of a penal statute. That statement and that opinion he had recanted. He had substituted another process (No. 2, as it was called); but he (Mr. Salamon) would warn him not to accept it without careful examination. He considered it to be as full of error as the abandoned No. 1 process.

Not being able to speak as a medical man, he could not discuss at length the President's contention as to the nutritive qualities of hop extract and the irritating qualities of quassia; but he did not think the position taken up was quite supported by the authorities upon the subject. The President has quoted from the older school of materia medica men, and had entirely ignored their greatest living authority, Lauder Brunton. By reference to his well-known book, he would find quassia described as a pure bitter stomachic tonic, the action of which is not thoroughly understood, and he would also find hops relegated to the category of hypnotics. Lauder Brunton further said that the combination of hops with lettuce in the form of a supper, consisting chiefly of beer and salad, had sometimes a very marked soporific action.

To use the President's own expression, the key-note of his argument was that the bitter principle of the hop was a glucoside. Hence, in the decomposition of the glucoside, the glucose was available as a food. He hoped it would not go forth that the Society approved that statement; for it did not embody the views generally held. The President had suggested that his case must stand or fall by that assertion. In that event he did not think there would be much difficulty in making it fall. The authority of Dragendorf upon such questions would scarcely be denied, and he supported Isleib (see "Plant Analysis," p. 147) in his confirmation of the view that it was not a glucoside at all.

Next, with respect to the statement that basic acetate of lead precipitates the bitter principle of the hop, it is curious that Mr. Allen and the President should entertain such opposite views upon a question so vital to the process. In the June number of the ANALYST, 1887, Mr. Allen said in his paper upon the detection of hop substitutes: "Several writers on the subject state that on precipitating a beer with basic acetate of lead the hop bitter is precipitated, and hence, if the concentrated filtrate still have a bitter taste, the presence of some hop substitute is certain. This difference seemed to me so important that I have very carefully investigated it, and find it perfectly in accordance with the fact." If this statement were contrasted with that of the President when before the Select Committee, the discrepancy would at once become apparent. Sir Henry Roscoe asked: "Are you aware in what respect Mr. Allen's method differed from yours?" "Yes. I first of all boil the sample with basic acetate of lead. Then, of course, I filter off the precipitate of the lead with such bitters as go with the lead. Then I acidulate my filtrate to get rid of my lead with sulphuric acid. Then I concentrate it into a small body, *when the sulphuric acid has the effect of discharging the fugitive bitter still further*." That must be taken as a confession on the part of the President that the bitter of the hop—the fugitive bitter—was not wholly precipitated by sub-acetate of lead. Other matters were, however, precipitated, which, according to the President's own statement, and according to his (Mr. Salamon's) own experiments, would render any legislative action based upon the President's process wholly abortive. The desire was to provide a method which would allow an analyst to swear to the presence of hop substitutes in the presence of hop extract. How could they say they had got such a process when according to the President's own admission, confirmed as it was by numerous experiments which he (Mr. Salamon) had made, chamomile would pass unchallenged? He begged leave to quote from the President's speech, recorded in the ANALYST for June, 1887. He said "he had worked on the question of hop substitutes some ten or twelve years ago, and he found no difficulty in distinguishing between the bitter of hop and the substitutes used for it. The method he found most useful was the precipitation by sub-acetate of lead, and there was no difficulty at all with the ordinary bitters, such as quassia, calumba gentian, chiretta, and wormwood all remaining in solution, whilst the bitter of hop, *and* *also the bitter of the chamomile*, which behaves like the hop, goes down and leaves the solution bitterless." But it must not be imagined that chamomile was the only substitute that could be used in defiance of legislation founded on the President's process. Among the bitter principles of chiretta, it had been ascertained (Pharmacographia, a History of Drugs, Fleckigür and Hanbury, p. 393) that ophelic acid occurs in the largest proportion, and that it produces an abundant yellow precipitate with basic acetate of lead. He would refer the President to a research by the late Dr. Griess upon the presence of cholin in hops (Trans. Chem. Soc.); he believed that cholin had been isolated in certain samples of beer, and he would ask the President whether it was not a bitter principle, and whether it was precipitated by basic acetate of lead. In such a case might not a wrongful conviction be obtained? But above all he would ask the President whether his experiments had covered the addition of caramel to hopped beer. It was well known that prepared caramel was used very generally in the manufacture of beer. Had the President isolated the bitter principle of caramel? He certainly could not regard it as a hop substitute, and was it, he asked, removed by treatment with basic acetate of lead. He (Mr. Salamon) had made experiments which showed him that it was not so removed, and further, it resisted the action of sulphuric acid. He suggested that the President had not made the experiment in question, and that with the important class of black beers containing added caramel, a wrongful conviction might be obtained every day according to the President's process. He (Mr. Salamon) did not believe that a satisfactory method would ever be obtained by using basic or neutral acetate of lead, with or without sulphuric acid. It would have to rest upon a different action altogether, and he submitted with confidence that he took up a legitimate position when he told the Select Committee that it was impossible by any known process to swear to the presence of added hop substitutes in beer in the presence of hop. He asked the meeting to confirm him in his view, and to state that the No. 2 process of the President would be less efficient than the No. 1, because the basic hydrate would precipitate all acid principles, many of which were bitter. A further weakness was apparent in the President's process. It was most important. The concentration of the beer after removal of the lead, and the continued boiling with sulphuric acid, might, and he thought would, result in the destruction of several of the bitter principles they were most anxious to detect. They found, for instance, that gentio-picrin was decomposed by dilute mineral acids into glucose and a neutral principle. What would be the result of a concentrated acid in such a case?

The President had apparently laboured under the misapprehension that the bitter principle of the hop was the same in every kind of hop. Any brewer would tell him this was not the case. Had he ever worked with foreign hops, notably with Californians? He asked the question because it seemed to be an offence to use foreign hops in Kent, where the President had his laboratory.

Next he came to the influence of brewing waters upon hop extract. It was well-known to those who had really studied the question that the bitter of the hop varied greatly with these conditions, and no process would be worthy of consideration which did not take this into account. He therefore asked the President whether he had experimented, as he, Mr. Salamon had done, with waters derived from different geological formations. For example, had he tried waters from the coal formation, the carboniferous limestone, the marls in the sandstone, the millstone grit, or the dolomite? He did not think he had, and in that case he was not justified in speaking with dogmatism as he had done before the Select Committeee.

The attitude adopted by Mr. Allen in this matter certainly required comment, and he could not pass it by unnoticed. When Mr. Allen went before the Committee, his attention was drawn to the fact that he had recanted his process published in the ANALYST. He admitted it was so, but said that notwithstanding he was prepared to give evidence on oath as to the presence or absence of hop substitutes in beer. On being pressed by Sir Henry Roscoe, he admitted that he had made no fresh experiments to support his changed views, and that he had only conceived them a couple of days before giving evidence.

It was essential that this process should be conducted upon half a pint of beer, otherwise it would not have conveniently brought itself within the scope of the Sale of Food and Drugs Act. Dragendorff required two litres; Mr. Allen, in his process, published in the ANALYST for June, 1887, required one litre, but in his evidence before the Committee he stated his ability to make the test upon half a pint. He, Mr. Salamon, challenged his ability in this direction He had repeated Mr. Allen's experiments, and could not get the same results. With regard to the question of quantity of beer, he would call attention to the fact that the hop extract did not amount to 5 per cent. onthe total solid matter in the beer, so that, in experimenting upon half a pint, there would not be more than 15 grains. Replace, say, half of this by hop substitutes, and there would not be more than about 7 grains of hop bitter and a considerably less quantity of the substitute to work upon. They must not lose sight of that question. But in the experiments which Dr. Bell made, and which he detailed to the select committee, he worked upon beer artificially bittered with quassia to the extent of six pounds to the barrel.

MR. HERON said he had for some years past given a good deal of attention to the subject of hops and their constituents, with a view to devising a means whereby their real marketable value might be determined by chemical analysis; and, in doing so, the bitter substances contained in hops had, as a matter of course, come under his notice. Since Mr. Adams had given his evidence before the Hop Industry Committee at the House of Commons, he (Mr. Heron) had repeated many of his old experiments, as well as tried new ones, in order to test the accuracy of the process mentioned by Mr. Adams in his evidence, and he had come to the conclusion that it was perfectly impossible, with our present knowledge of the subject, to precipitate out of solution, or to remove by any known means, the whole of the bitter substances of the hops. He had tried Mr. Allen's process as well as that of Mr. Adams. He had also used barium hydrate, as Mr. Adams had recommended in what he called his No. 2 process; and, although working in the most careful manner, he found all those methods to be practically worthless. In these experiments he had not confined himself to one kind of hops only, but had tried old as well as new hops, and foreign as well as English; and, treating them as he would, he still found in the final result, the bitter taste of the hop persistent.

No doubt Mr. Adams was well aware of all the work which had been done on the constituents of hops, and, if so, would know that there were at least three bodies contained in hops, which possessed a most distinct and decidedly bitter taste; one of these might possibly be precipitated by lead acetate, but there was evidence that the other two were not, and apparently could not, be removed by any other reagent either.

With regard to the whole subject they were simply in this position now. They knew that hops contained several substances possessing bitter properties, and until a thorough and exhaustive examination of different varieties of hops, of all ages and from all countries where hops are grown, had been made, until those bitter substances contained in them had been separated, and until their exact behaviour to chemical reagents had been determined, no one could positively assert anything with regard to those bitter substances or lay down any rigid rules for their detection.

Mr. Heron was rather surprised that Mr. Adams should refer to such antiquated

thorities as Pereira and others in connection with the stupefying effects or otherwise
f hop extracts. He for one preferred to consult the works of such men as Lauder
runton, Stevenson, Griess, and Williamson, men who had gone into this question in a
oroughly scientific manner, and who had shown beyond a shadow of a doubt that a
rcotic principle did exist in hops, and which possessed, when in a very concentrated
rm, properties similar to those of other well-known narcotics; but he considered there
as hardly any occasion to go beyond one's own observation to prove this. Every one
ere would doubtless agree with him that new whiskey contained an appreciable
ount of fusel oil and other of the higher alcohols, and that a glass or two of such
hiskey would contain more of these higher alcohols (to which Mr. Adams ascribed the
upefying effects of beer) than probably a gallon of strong ale; but what was the result
hen people indulged in an immoderate quantity of either? Simply this, that where
hiskey was indulged in to excess an exhilaration of spirits was produced, which, in the
ter isle, had, in days gone by, led to many a faction fight and row; but the very
posite was the case with people drunken with beer, they became drowsy and stupid
owing very little inclination for fighting or for anything else, and he considered it was,
erefore, merely begging the question for Mr. Adams to say that the stupefying effects
beer when indulged in to excess came from the alcohol contained in it and not from
e hops.

There was another matter he would like to refer to, namely, with regard to the
tion of various brewing waters upon hops. There was no doubt that some of the mineral
nstituents of such waters exerted a greater extractive influence than others, as he had
und by actual experiment. Again, the malt used in brewing would play some part,
cording as it contained more or less lactic acid. All these points should be considered
conducting experiments upon hops and in the conclusions derived therefrom.

He regretted very much that a paper on such a subject had been read at such a
me and under such circumstances. Mr. Allen had read a paper on a similar subject
o years ago, and in it referred in unfavourable terms to the use of sulphuric acid in
ecipitating the excess of lead in the filtrate. Mr. Adams was there and took part in
e discussion which followed. He had then an opportunity of referring to his own
ocess, in which he had placed so much confidence, but he allowed the opportunity to
ss, and he (Mr. Heron) would like to ask Mr Adams why he did not then bring his
ocess forward for the benefit of his brother analysts, instead of on the present occasion,
the eleventh hour, when the secret could no longer be kept.

Very careful and numerous experiments ought to be made and tested under all
nds of conditions, before any method upon which must hang such an enormous amount
responsibility, and upon the result of which a person could go into a court of justice
d swear that such and such was the case, was made public.

MR. HEHNER said that by raising side issues, as had been done by the two previous
eakers, and suggesting that various kinds of brewing waters, or hops from different
untries might yield different results, the main question would be hopelessly obscured.
course these matters would ultimately have to be studied, but for the present they
d to deal with the comparatively simple question: Is there a possibility of distin-
ishing between the bitters of the hop and of hop substitutes, and does the process
oposed by Dr. Adams effect such a distinction, or does it not? They had on the one
nd the definite statement of Dr. Adams, that it was capable of doing it, whilst on the
her they had an equally emphatic denial. He could not help thinking that the gentle-
en who were thus at variance could not have operated in precisely the same manner.
nall differences of manipulation might be at the bottom of this issue of opinion, and
thought it would have been well if Mr. Salamon and Mr. Heron had first com-

municated with the author of the paper to make sure they approached the matter upon the same basis.

During the most able speech of Mr. Salamon he had received the impression, as if that gentleman had been more anxious to reach those outside that meeting who were interested on the question, than to discuss for the benefit of the members of the Society. Public analysts had no political interest in the hop question, but it behoved them, in view of the possibility that they might be shortly called upon to distinguish between hop beers and otherwise bittered beers, to attempt the solution of this difficult question.

He quite agreed with Mr. Salamon as to the magnitude of the difficulties to be overcome. The number of bitter substances which might possibly be used as hop substitutes was so great, and their chemical value so widely different, that there was no possibility of classifying them by a few simple reactions. Only a short time ago it was believed that the precipitation with lead acetate sharply separated hop from other bitters. This belief proving more or less erroneous, Dr. Adams had now introduced a further mode of distinction, namely, the treatment with acids.

As a rule, glucosides on treatment with acid, yielded sugar and a bitter substance, whilst indifferent, non-glucosidic bitters were little affected, but in some instances the products of decomposition of glucosides were not bitter; thus colocynthin gave colocyntheine which was perfectly tasteless. Whatever might be the chemical nature of the hop bitters, he was of opinion that Dr. Adams had given them the means, by the successive treatment with lead and with acids, of narrowing the question and of enabling them more nearly to classify the bitters. For this they should feel grateful, even if it turned out that these views by themselves were not sufficient to sharply distinguish hop from all other bitters.

Dr. Johnstone said that in a paper of his on this matter about eighteen months ago he mentioned about the washing of the hops.

Mr. Cassal said that specific information was required as to the steps taken to watch over the hops in the hop-ground. He regretted that such experiments as he had been able to make in reference to this matter had up to the present been very few, and he was bound to say that hitherto he had not succeeded in removing the "bitter" in the manner directed. He recollected speaking to the Chairman (Dr. Sykes) upon the question, and his (the Chairman's) suggestion that the hops might have been watered with a solution of quassia. It was very evident that something far more definite and reliable than what they were at present in possession of, was required. From the point of view of a public analyst, who might be called upon to subject himself to cross-examination in the witness-box, the matter was far from being in the position that it should be. He did not think, like his friend Mr. Heron, that it would be for all time impossible to detect the presence of hop substitutes in beer; but he was disposed to think that the position adopted by Mr. Salamon before the Committee was upon the whole justifiable; it was, at any rate, safe. The Sale of Food and Drugs Act in its passage through Parliament had been largely affected by persons interested in adulteration in some form. The Act was exceedingly and unnecessarily cumbrous, and compared very unfavourably with certain Continental laws. The reasons for this were perhaps not far to seek. There was no earthly reason why public analysts should be compelled to adapt their processes of beer analysis to the examination of such quantities as half-a-pint, except that the present cumbrous law made it necessary for Inspectors to ask for small quantities in order not to arouse the suspicions of vendors prior to a purchase. There was no valid reason why an Inspector should not have power to seize any reasonable quantity of beer or of any other article which might be required for the purpose of proper analysis. The whole system of sample-taking appeared to have been devised for the benefit of adulterators.

It appeared to him (Mr. Cassal) that a little too much had been made by some peakers of the harm that hops might do to a consumer as compared with other things. hey were asked to believe that the brewer was using hop substitutes in order to provide e public with something that was better for them, and in the profoundest anxiety to rve his fellow man. Every one might not see it in that purely philanthropic light. f an infusion of malt and hops was generally recognised by the public as beer, and if ey wanted it they had a right to get it—and to get it free from adulteration; but it as first necessary that what was to be understood by beer should be clearly defined.

Owing to the lateness of the hour, MR. ALLEN and DR. ADAMS confined themlves to a few short remarks. These gentlemen have since furnished us with e following replies:—

MR. ALLEN says:—It is a curious and interesting fact that Mr. Salamon nd Mr. Heron have never come forward with any hint or suggestion, and have ot attempted to help the Society in any manner, though they have been ertile in prognostications of failure on the part of those who are attempting to olve an admittedly difficult problem. It appears from the speeches of Mr. alamon and Mr. Heron that these gentlemen have made many thousands of experiments n the detection of hop-substitutes, and Mr. Heron goes so far as to say that not only as no process been devised in the past, but that it is absolutely impossible that any rocess can be devised in the future by which hop-substitutes can be detected in beer. t is very curious that, after taking so much trouble, neither Mr. Salamon nor Mr. Heron has placed a single one of their experiments on record, or laid before public nalysts their reasons for believing the problem to be insoluble. It was said that Mr. Adams' paper was inopportune, but such an opinion comes from Mr. Salamon with a ery bad grace. Is the paper inopportune because it is now of practical interest; r because it is exceedingly inconvenient to Mr. Salamon and his friends, now that Parliamentary Committee is sitting to consider whether the unacknowledged use of op-substitutes shall be permitted? Mr. Salamon says that the President should have ead his paper *before* he gave his evidence; but where was the opportunity? There as no meeting of the Society in the previous month, and two months ago the question ad not arisen. And how comes it that Mr. Salamon has done that for which he blames he President? Mr. Salamon has taken the extraordinary course of going before the Committee, and in the absence of and without consultation with me has undertaken to nterpret—or rather misinterpret—views held by me. Mr. Salamon has thought proper o criticise my position in the matter, but apparently has never taken the trouble to master what I have written on the subject.

The use of acetate of lead has been recommended by a number of chemists as apable of precipitating the hop bitters, while leaving all or nearly all probable substiutes in solution. Dragendorff was apparently the first to suggest it, and he gave n elaborate scheme for detecting the substitutes in the filtrate. As stated in my aper published in the ANALYST for June, 1887, the distinction is referred to in Vincent's "Chemistry Applied to the Arts and Manufactures," in Wynter Blyth's "Analysis of Food," and forms an essential part of Ender's method of detecting bitter substances in beer, so that there is a very general consensus of opinion as to the value of the method. Having all this evidence before me when making the experiments which formed the foundation of the paper already referred to, I contented myself with a limited number of experiments in confirmation of this hitherto unquestioned proposition. Experience has shown that I was too hasty in this, and that the lead process as described by me in the spring of 1887 is unsatisfactory as a sorting test in the case of particular samples of hops; but in face of the unanimous dictum of previous workers this could not possibly have been foreseen. Indeed, a few minutes before the meeting at which my paper was read, Mr. Adams informed me that precipitation by lead was also the principle of the method he had successfully employed for the same purpose. But as my paper clearly indicated, I never intended the treatment with lead acetate as anything but a clarifying and sorting process, which was to be followed by extraction of the bitter principles with chloroform and ether, and the application of other tests to the principles so extracted. I contend that if this be done any traces of hop bitter which escape precipitation by lead are perfectly distinguishable from other bitter principles, and hence I was justified in saying that I was prepared to swear to the presence of these substitutes in all or nearly all cases, although if applied in the manner originally described by me the lead process did not make so sharp a separation as I had supposed. Hence, I have by no means "recanted" my previous expression of opinion that it is perfectly feasible to distinguish hops from hop-substitutes, with perhaps one or two exceptions, and I am quite prepared to detect and identify quassia and several other hop-substitutes in a perfectly positive manner without relying on the lead process at all. But much of the value of the lead process undoubtedly depends on the way in which it is used, a fact which explains the conflicting experience respecting it. For instance, I have stated before the Society at a previous meeting that a particular sample of hops, the bitter of which was not wholly removed by the ordinary treatment with acetate of lead, gave a perfectly satisfactory result when the lead was removed by sulphuric acid instead of by sulphuretted hydrogen. Whether such a change in the manner of removing the lead would be successful in every case I am not prepared to say. Then Mr. Heron said that the President adopts a method of treatment which was "condemned" by me in my paper published in the ANALYST for June, 1887, and he explained that he referred to the use of sulphuric acid for removing lead. But what I actually did say was "I *prefer* sulphuretted hydrogen to sulphuric acid or a sulphate, as the lead sulphide seems to carry down a notable quantity of colouring matter." This "condemnation" was certainly of a very mild character.

Dr. James Bell, in his evidence before the Committee, stated that the weak point of the lead process was that the extractive matter of beer was apt to prevent the complete precipitation of the hop-bitter by lead, and that in the absence of that extractive matter the precipitation of the bitter was complete. In answer to a further question put to him at my instigation, Dr. Bell definitely stated that if the lead treatment were repeated upon an extract obtained by ether or chloroform in the manner indicated in my paper before the Society of Public Analysts, the remaining trace of hop-bitter was removed, and that in that manner it was possible to make a perfect separation of the bitter of the hop and the bitters of hop substitutes. So far Dr. Bell's evidence is positive. His evidence as to the detection of the substitutes is discounted, as is that of Mr. Bannister, because they used such a proportion of quassia and other hop substitutes as would render the beer quite undrinkably bitter. I have operated on what I think a better plan, which is to take a mild beer containing no bitter unremoveable by lead acetate, and to add to such beer sufficient of the hop substitute to render it agreeably bitter. When such beer is subjected to the lead or other sorting process, there is no mistaking the bitter taste of the hop-substitute. It can be recognised not simply on half a pint of the beer, but on a mere fraction of that amount; in fact, there is much truth in the suggestion of Mr. Hehner, that my attempts to improve the methods of detecting hop-substitutes have rendered the tasting process too delicate. The very fact that Mr. Heron and Mr. Salamon seem to doubt the possibility of getting a bitter extract *from* half a pint of beer shows that their experience is of a very peculiar kind. They ve only got to *try* the process *once* on a beer bittered with quassia or chiretta, and eir tongues will proclaim conviction. Mr. Salamon quotes Hanbury and Flückiger's Pharmacographia," as an authority for the statement that of the bitter principles of iretta, ophelic acid, occurs in the largest proportion, and that it produces an abundant llow precipitate with basic acetate of lead, and he adduces this fact in support of his ggestion that chiretta would escape detection by Mr. Adams' process. But Mr. lamon omits to state that chiretta also contains chiratin, a principle which some servers consider the most characteristic constituent of the plant. Flückiger describes iratin as "intensely bitter," and Kemp actually employs basic acetate of lead to ecipitate other matters and obtain it in a purified condition. Anyone can be excused r not being acquainted with the chemistry of chiretta, unless, like Mr. Salamon, he dertakes to criticise the work of others, and then he should at least take care to scribe their researches accurately.

Mr. Salamon correctly refers to Dragendorff as a very high authority on the isola-n and identification of plant-products, though in doing so he apparently overlooks e fact that Dragendorff has published elaborate schemes for the separation and identi-ation of bitter principles, based primarily on precipitation by lead, and the use of miscible solvents. But Mr. Salamon should at least quote his authority correctly. It not correct that Dragendorff ("Plant Analysis," p. 147) supports Isleib in the view at hop-bitter is not a glucoside. What Dragendorff states is that Isleib confirms the atement of some unnamed observer that hop-bitter is not a glucoside. Mr. Salamon ould be more careful.

Again, Mr. Salamon correctly states that by boiling with dilute acids, gentipicrin is lit up into glucose and a neutral principle, but he omits to state that this neutral inciple is *bitter*! I believe no one but Mr. Salamon can have understood the resident to propose operating so as to produce concentrated sulphuric acid.

I am certainly inclined to share the doubt as to the glucosidal character of the p-bitter, but I consider that the question had no practical bearing on the point at sue. The point, so far as Mr. Adams' process is concerned, is whether it is possible by treatment with acetate of lead, perhaps supplemented in some way, to remove the tter principle of the hop, while leaving the bitter principles of the usual hop sub-itutes untouched. Mr. Adams says he can do that, with the exception of chamomile, d it is no use for Mr. Heron to come forward and say that it is impossible; unless he kes the ordinary course of laying the results of his experiments before the Society in tail, and describing exactly his method of operating, so that we may be able to trace e cause of his failure. It is not contended by me, and I do not suppose it is con-nded by Mr. Adams, that it would be possible to declare in *every* instance that a beer ntained hop substitutes when such might be present. Mr. Adams admits, and stated ng ago, that the bitter of chamomile behaves like that of the hop with acetate of lead, d I have no doubt that, should a law be passed making it illegal to use hop-substitutes ithout declaring the fact, chemists would be found who would use all their ingenuity circumvent the Act by discovering other bitters which would escape detection by any ocess which might be devised. Of course we have always to take account of that nd of action. Milk and butter are adulterated in a scientific manner according to the rections of professional chemists, and we may be sure the same will be done with beer. ut it is not necessary that public analysts should be able to affirm positively that a er is unadulterated, as Mr. Salamon seems to assume. What is required is that public alysts shall be quite certain that they are not liable to condemn, on wrong grounds, a er bittered solely by hops. Mr. Salamon suggests that choline is a bitter principle, curring in hops and present in beer, not precipitated by lead, and liable to be mistaken for a hop substitute. But what is the truth? In all the descriptions of choline I have been able to find, not one author, except Griess, refers to it as a *bitter* substance. Even in the very paper by Griess (*Jour. Chem. Soc.* XLVII., 298), quoted by Mr. Salamon, and with which he must, consequently, be well acquainted, choline is merely described as having a "caustic, *somewhat* bitter taste." Its characters preclude its extraction from aqueous liquids by ether or chloroform, and Griess could not obtain more than 0·02 per cent. from hops. At the rate of 6 lbs. of hops per barrel, this means 2 milligrams. of choline per pint of beer, and Mr. Salamon suggests that the presence of this proportion of a "*somewhat* bitter" substance, natural to hops, and not extracted by ether or chloroform, would invalidate the detection of hop-substitutes! But, above all, Mr. Salamon lays stress on the difficulty he fears would be caused by the presence of added caramel in black beers, and he gravely asks if the President's experiments have covered such additions. Does Mr. Salamon seriously suppose that any public analyst would be likely to condemn a black beer under such circumstances. The lead-sorting test was never intended to be applied rigidly in such a case. It should, however, be within Mr. Salamon's cognisance that the books describe the bitter constituents of caramel as precipitable by lead acetate and other metallic salts. And if Mr. Salamon will take the trouble to *try* the effect of adding basic acetate of lead to a solution of caramel, he will find that by far the larger part of the bitterness is precipitated, and if he will then extract the concentrated filtrate with chloroform, and repeat the whole treatment on the chloroform extract, he will find that every trace of bitterness has been got rid of. If he uses neutral lead acetate he will probably find the bitter to be wholly precipitated by the first treatment. Hence, Mr. Salamon need not fear the confusion of caramel with hop-substitutes.

Mr. Salamon states that every brewer is aware that the bitter principle of the hop varies with the district in which the hop is grown. That the amount of bitter principle and the flavour are liable to variation will be intelligible to all; but Mr. Salamon appears to suggest that the chemistry of the hop varies with the initial of the name of the county or country in which the plant is grown, and that if the hop has been grown on a sandy soil, acetate of lead will not be likely to produce the same effect on an infusion as if the plant has been grown on a cretaceous soil. The first impression produced on hearing such contentions is that they are ridiculous; but if Mr. Salamon puts them forward seriously, he should at least back them up by experiments, showing the influence of the nature of the soil on the character of the bitters in the hops grown thereon. Such a paper would be a valuable contribution to the literature of plant-chemistry, but until it is produced it is impossible to treat Mr. Salamon's contentions seriously.

Then, again, Mr. Heron came before them and said that water containing 15 grains per gallon of sulphate of sodium extracted more of the bitter principle of the hop than water free from that saline constituent, and he wished them to believe, not only that more of the bitter principle was extracted, but that it was different in character. Mr. Heron said that he had proved that it was different in character because it was not precipitated by acetate of lead; but, unfortunately, Mr. Heron had apparently failed in precipitating the bitter of any hops by acetate of lead, although many chemists had employed that reagent for the purpose.

Mr. Salamon says he has examined waters from various geological formations, and asserts that it is well known to those who have studied the question that the bitter of the hop varies materially according to the origin of the water. Does Mr. Salamon mean to say that it is well known and that he has proved that the geological formation from which the water used for infusing the hop is derived affects the power of lead acetate to precipitate the bitter principles? If so, where is a record of this "well-known" experience to be found?

In conclusion, we must admit that the problem in question is undeniably a difficult one, but the inherent difficulties are enormously increased by the action of a certain section. Chemists seem to be divided into two classes; those who never communicate any of the knowledge they possess, and are never to be found helping their brother chemists, though they are always ready to throw cold water on their efforts; and another party who feel it their duty to help their brother chemists by laying before them such information as they have, and who, perhaps, sometimes err, as I myself have done, by too much readiness in that way. In my opinion, the Society are very much indebted to the President for the paper he communicated. I do not suggest that we are in a position to rely on the modified method proposed, even as a sorting test, without due trial of it ourselves under various conditions. It is our duty to investigate the bearings of every process submitted to us, but the Society are indebted to any member who will bring before them his experience on any subject, so that it may be duly tested and verified by each member interested in the kind of work in question. But if we are to wait until we have examined samples of hops from every county in England, and every country in the world, grown on every kind of soil, and brewed with water from every geological formation, as Mr. Salamon suggests we ought to do, legislation on the question must be deferred till the Greek Kalends.

Mr. ADAMS says.—I should like to say the following in answer to some of Mr. Salamon's remarks:—

In the first place, my evidence before the Select Committee was not "given in the name of the Society," but on my own individual responsibility, and I had no alternative but to take the very first opportunity for placing my views before the Society, because I had pledged myself to do so. Moreover, so far as the Select Committee is concerned, the inquiry relating to the science of the subject "is not closed," but, on the contrary, has been specially kept open to afford an opportunity for me to make known my views in detail, so that they might be put to the test. As for misleading the Committee, or recanting my opinions, that is all nonsense. The most that can with truth be said is that my opinions have developed, and the House of Commons' Committee well know how prompt my action was to acquaint them with the discovery which I made and was the first to announce, viz., that there is a difference in the behaviour between new and old hops under chemical treatment. I devised a method (No. 1 Process) successful for distinguishing between the bitter of new hops and the bitter of quassia and its allies. Also another (No. 2 process), equally successful for the same purpose in the case of old hops. If I had not taken the course that I have taken as regards making known these things, I should have laid myself open to much blame.

Mr. Salamon says I have *suggested* that my case must stand or fall upon the *assertion* that the bitter principle of the hop is a glucoside. Such is not my suggestion, and I have asserted nothing of the kind. The suggestion is Mr. Salamon's, not mine. My assertion is that the facts fit in very well with the suspicion that a part, or the whole, of the bitter of hop is of the nature of a glucoside, and I beg to refer Mr. Salamon to the recent researches of Greshoff (*Allge. Brauer u Hopfen Zeit.*), which have proved my suspicion correct, by showing that a portion is a glucoside.

Mr. Salamon has confused two distinct and independent things—the facts and the explanation. The facts are facts; the proposed explanation is a theory. The validity of the process stands by the facts; the explanation must take its chance, and stand or fall with the theory. As respects Mr. Salamon's contention that because chamomile would escape detection by this process therefore the process must be abandoned, was there ever an argument more ridiculous? It is quite unreasonable to expect any one single test to shut out every possible resource for adulteration. Experience of the working of the Food and Drugs Act is all against such an absurd contention. Objection might just as well be made to a process for detecting cotton-seed oil in butter because it was not fitted for the detection of beef stearin. For all practical purposes quassia is *the* thing that is used as a hop substitute. Chamomile was tried in 1882, but found quite unsuitable because of the large amount of volatile oil it contains.

Regarding the complaints of Mr. Salamon, and also Mr. Heron, that I had only quoted from the older school of *materia medica* men, thereby implying that I had not referred to any modern authorities, that is not true. Quotations were given from four sources, two of which, Pereira and Neligan, were among the greatest authorities of the past generation, and Garrod and Ringer are living authorities of the highest rank in the present generation.

Mr. Salamon had been pleased to designate Lauder Brunton as the greatest living authority. I will not stop to question his right of doing so; but I will only say that if such be really the case, more is the pity that Mr. Salamon should have given incomplete and therefore incorrect and misleading quotations from Brunton's writings. "By reference to his well-known book," Mr. Salamon must have observed when speaking of quassia he says, "In small doses it increases the appetite; in large doses it acts as an irritant, and causes vomiting." Again, to understand properly the passage relating to the supper of hops and lettuce, the context must be read, which runs as follows:—"Lettuce certainly seems to have a hypnotic action, but how much it depends upon the juice and how much upon the mechanical effect of the indigestible fibre of the lettuce upon the stomach in drawing blood to it, it would be hard to say. Hops are said to be hypnotic *in their combination with lettuce*. In the form of a supper, consisting chiefly of beer and salad, it has sometimes a very marked soporific action." So that, to produce the soporific effect, besides the hop this famous supper requires the *alcohol* of the beer, *the juice and also the indigestible fibres of the lettuce.*

And to show how utterly sceptical Dr. Lauder Brunton is of the soporific properties of the hop, see what he says in the very next following paragraph:—"A hop pillow is sometimes used in sleeplessness of fever, but its use is probably due not to the action of the volatile principles of the hop" (the only part to which soporific properties are usually attributed), "but to the mechanical elasticity and softness of the pillow."

Surely Mr. Salamon is to blame for want of candour giving us these quotations in full, seeing that they are necessary for the object he expresses himself so anxious, viz., that there shall be "*no unintentional misleading.*"

THE ANALYST, Vol. XIV., for 1889.

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THE ANALYST.

AUGUST, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON DYED SUGAR.

By Charles E. Cassal.

(*Read at Meeting, July*, 1890.)

tention has recently been directed to dyed sugar in consequence of a number of
s of moist sugar purchased in the ordinary way by the inspectors of one of the
ts for which I am public analyst having been submitted to me. I am desirous of
ng the matter before the Society in its relation to the official work of public
ts, and of calling special attention to it as being one which appears to me to be of
erable importance, affecting general public interests as well as those of a large

Some two or three years ago it became known to many of us, and to persons in
de, that certain firms had introduced sugar-crystals which had been artificially
ed. If I am correctly informed, it was not at first known in the trade that most,
all, the sugar that was so introduced was beet sugar and not cane sugar; and I
I am right in stating that the original name given to these products was that of
cane crystals. The present recognised trade name is "yellow crystals," and I do
ppose it will be denied, that the greater portion of the yellow crystals of commerce
of artificially-coloured beet sugar.

he different samples submitted to me officially were plainly not the products of
me process of treatment, and therefore probably not of the same firm. They con-
of sugar crystals possessed of various shades of golden-yellow, approximating in
ases to orange. Since examining these samples I have obtained authentic specimens

of the dyed sugars of different firms, and have been engaged for some time in experimenting upon them. Somewhat varying degrees of skill would seem to be employed in the preparation of the sugars. Some samples have been very skilfully coloured, while with others the work has been coarsely and roughly done. It will be found by examination with a lens that in the products of certain firms a number of crystals are entirely colourless and can easily be picked out, while with others the uniformity of colouring is far more perfect. In addition, large nodules of a bright orange colour are frequently to be found in what, I presume, may be called the more inferior products.

The particular sugar to which my attention was first directed was one the dye on which can be detected with the greatest ease. The sugar itself, or a solution in water, strikes a peculiar purple-pink colour on the addition of a few drops of concentrated hydrochloric acid. The colour is characteristic, and, if the dye is present in unusually small amount, is best seen when about 25 to 30 grammes of the sugar are just moistened with hydrochloric acid in a deep conical porcelain dish. On standing, the mixture becomes of a dark, reddish-brown colour. Acetic acid has a hardly-perceptible effect, but appears to slightly intensify the orange colour of the crystals. Sulphuric acid produces the same effect as hydrochloric acid, but the subsequent changes of colour are not properly perceptible owing to the charring that takes place. Although from the point of view of the public analyst it is a matter of little or no immediate importance to be able to detect the particular dye-stuff or dye-stuffs which have been used, so long as an artificial dye can be certified to be present, I may direct your attention to the fact that these reactions are very similar to those obtained with certain of the so-called Tropæolins, especially in some respects with those which seem to be known in commerce as "Tropæolin OOO" or "Orange I.," and "Tropæolin OO" or "Orange IV." Tropæolin OOO is the only one which I have examined which gives the rapid change of colour from purple-pink to dark-reddish brown when treated with hydrochloric acid; which change, as previously stated, was obtained with the samples of sugar now referred to. I have not, however, succeeded in satisfactorily dyeing colourless sugar-crystals with this substance by itself, the colour obtained being dissimilar from that upon the commercial sugar. Further, acetic acid, when added to solutions of Orange I. and Orange IV., does not produce any red or pink colour, but merely what appears to be an intensification of the orange colour. For these and other reasons, which I propose to communicate later, I considered that while the particular sugars referred to had probably been dyed with Tropæolins, it was likely that a mixture of these bodies had been used. I have now reason to believe, from information which has been afforded me, that this is what in reality had been done. It must be remembered that great confusion exists with respect to the application of these commercial names. My remarks here apply to substances which have been purchased in the ordinary way under these names.

Other dye-stuffs are, however, used for the purpose of colouring sugar-crystals, which do not give the reactions with hydrochloric acid, and it has therefore been necessary to devise means for showing their presence with sufficient reliability to admit of their being certified to. By applying the following simple process, I believe that any of the dyed sugars at present on the market can be sharply distinguished from those sugars which contain no colouring-matter foreign to sugar. About 100 grammes in most cases (less can be used if absolutely necessary), of the sample is washed in a flask with alcohol of 90° per cent.. This removes the dye in the majority of the cases which have come under my observation. In some cases several washings are necessary, in others one only. Treatment with alcohol of this strength removes the whole of the dye, leaving perfectly colourless crystals. I have found that it is advisable to use weaker alcohol with some dyed sugars, viz., from 75 per cent. to 80 per cent. The solution is filtered from the sugars, evaporated to dryness, again taken up with alcohol, and a skein of silk or wool (preferably slightly mordanted with aluminium acetate) treated with the solution, warmed for some time in the water bath, and subsequently well washed with water. The skeins are permanently dyed of a more or less marked yellow colour if a dye has been extracted by the alcohol from the sugar.

A sample containing only such colouring-matter as is natural to sugar, even by repeated washings with alcohol of 90 per cent., does not leave absolutely colourless crystals, and does not give a solution capable of permanently dyeing silk or wool.

One of the substances which has evidently been very largely used for the dyeing of sugar is the so-called phosphine or chrysaniline. Sugar dyed with phosphine can at once be detected by the foregoing process. As commercial phosphine is usually the nitrate or the hydrochloride of chrysaniline, no doubt it is one of these bodies that is used. Fibres dyed with phosphine are turned to a very characteristic pale greenish yellow by alkalies which is more intense than the colour on the fibres when the latter is slight, and acids slightly redden them. When the amount of the dye upon the fibres is slight, the application of alkali serves to "bring out" the colour. I have obtained these reactions in every case upon fibres dyed with solutions obtained from sugar suspected of being dyed with phosphine. Fibres dyed with extracts containing the colouring-matter which, for the sake of convenience, I will call the "Tropæolin dye," are reddened by mineral acids.

It is evident that great quantities of dyed sugar are sold in London and elsewhere. It is very generally sold as Demerara sugar, and not as "yellow crystals." Out of fifteen samples of sugar purchased recently in one of my districts as "Demerara," eleven were unquestionably dyed, and the adulteration was detected easily in each case by the processes previously described.

I may be allowed to quote here an extract from a report which I made upon the subject a short time back. "Purchasers are under the impression that they are being supplied with genuine cane sugar (such as "Demerara"), whereas they are in reality getting sugar crystals which have been artificially dyed. In this way beet-root sugar can be coloured and sold as "Demerara," and it is generally admitted that weight for weight, under ordinary conditions of use, beet-root sugar does not give the same "sweetening" as cane sugar. A natural product having been tampered with by admixture with an artifical dye, whatever the real object may have been in the preparation of these sugars, it is obvious that the proceeding enables one article to be substituted for another." I am reliably informed that when "Demerara" is asked for on the market, *cane* sugar from Demerara is expected. The market price further, is stated to be from 17s. to 18s. per cwt. for Demerara, and for "yellow crystals" from 12s. to 13s. per cwt. A somewhat remarkable statement has been made with reference to my intention of certifying artificially dyed sugar sold as Demerara as adulterated; namely, that cane sugar produced in the West Indies is dyed there with the same substances as those previously alluded to. This is merely an attempt to create a new definition for Demerara sugar. It might almost as logically be urged that if growers of coffee were to take to mixing chicory with their product before exportation, we should no longer be able to regard chicory in coffee as an adulterant. If the case is not strictly parallel the device is the same. It may, on the other hand, justifiably be contended that the sugars dyed as I have described are adulterated, for they can be certified to have been produced by dyeing colourless sugar crystals yellow, with an artificial organic colouring matter not natural to sugar.

Another aspect of the subject in reference to which I may again quote from my report is this: "Many substances used as dyes, and closely related to the classes to which those found belong, are known to be of a poisonous nature, while the action of others, when ingested, is unknown, while others again are asserted upon very insufficient and unsatisfactory evidence to be non-poisonous. Apart, therefore, from other considerations, these cases afford an illustration of the impropriety of tampering in such a way with articles of food without, at least, an open statement of what has been done. Although in dyeing an article of food the weight of the dye-stuff used must be exceedingly small as compared with the weight of the substance dyed, it must be admitted that the proper course of action in the present state of knowledge on the subject, is, as far as possible, to prevent the use of dye-stuffs of the general character indicated for colouring articles of food, if for no other reason, in view of probable and perhaps dangerous abuse."

In giving official certificates in these cases it will be tolerably obvious that it is undesirable, as well as unnecessary, to make too specific statements as to the precise nature of the dye or dyes that may have been used. The statements that may be made by experts in giving evidence in matters of this kind very largely depend upon the standpoint from which they are viewed. If, for instance, a definite statement were made as to the precise character of a complicated dye-stuff present in very small quantity in sugar, or anything else, it would no doubt be perfectly easy and conscientiously justifiable for the gentleman who had prepared the mixture to swear in the witness-box that the compound certified was not present at all, if the compounds were not in every respect, physical as well as chemical, in composition, as well as in constitution, identically the same in the opinion of every chemist of repute, or even of non-repute. Whatever the standpoint of the chemist consulted by the manufacturer may be, and I fully admit the propriety and value of such consultation, when professionally legitimate and publicly useful, the position of the public analyst is clearly defined. It is his duty to the community which he is appointed to serve to advise them of the existence of adulteration when it comes before him, and to do so without fear and without favour. That the dyeing of sugar in the manner indicated, and its sale as genuine natural sugar, and the application of the process for the purpose of enabling one thing to be palmed off for another, are adulterations, and, therefore, offences against the law, I, for one, have not the faintest doubt. The public have every right to expect that natural food products shall not be coloured with artificial dyes without an open statement of the fact. I desire to express my thanks to my assistant, Mr. Gerrans, A.I.C., for the help which he has afforded me in carrying out the necessary experiments.

Discussion.

Dr. Dupre said he had repeatedly had sugars submitted to him artificially coloured, but never as public analyst, and he should feel doubtful himself as to certifying sugar as adulterated, simply because it was artificially coloured, unless it was proved that the colouration was for the purpose of deceiving and enabling it to be sold as Demerara sugar. He would like to hear the opinions of other analysts as to whether the mere fact of sugar being coloured was to be taken as an adulteration. Butter was often coloured, so was milk, but many analysts do not consider the colouration of butter to be an adulteration. It had been a long-continued custom, and the public liked to see their butter yellow. If an artificial butter was coloured with a view to its being passed off as a genuine butter that was a different matter, but the mere fact of its being coloured was not an adulteration.

He quite agreed with Mr. Cassal that a public analyst was not bound to state the exact composition and character of what he supposed to be the adulterant, for if it came to that, and they should have to put the exact composition of such and such a substance, and if they linked together wrongly the oxygen and hydrogen, and were then liable to have their certificates set aside, the sooner they gave up the better. All that the public analyst could be expected to do was to say that this particular sample, say of sugar, is artificially coloured. If he can go a step farther and say that the colouring matter is so and so, that would be an advantage, but otherwise it would be a most dangerous thing for the public analyst to bind himself down to a particular composition. He did not think that the Act at all required the public analyst to do that.

He would like to ask whether Mr. Cassal had submitted these colouring matters to spectroscopic examination, because so many of them could be identified with a tolerable degree of certainty by means of the spectroscope.

He had also found that artificial colouring matter added to sugar was almost as easily distinguished by the action of water, natural sugars colouring water very slightly only, whereas artificially-coloured sugars imparted a marked colour, like chicory compared to coffee.

The colouring matter added might be the same kind as that of the natural sugar, but there was this striking difference—when water was added to the sugar the artificial colouring matter was dissolved off readily and left colourless crystals, the natural sugar remained the same colour.

Mr. A. W. Stokes said that the public analysts were on the horns of a dilemma. It is well-known that almost all Demerara sugars contain about one-third of a grain of chloride of tin per lb., while most other sugars were artificially coloured with aniline colours in minute quantities. So that, from Mr. Cassal's view of the subject, few moist sugars would pass as unadulterated.

Mr. Stokes gave the opinion of Mr. F. J. Scard, the analyst at Demerara of the Colonial Company, who contends that the sale of fictitious "Demerara sugar" should be stopped under the Merchandise Marks Act. Mr. Scard holds that Demerara sugar keeps at the head of the market because of its retaining the natural aroma, tint, and flavour of the sugar-cane. Over-refining gets rid of these, and produces a less palatable article. He contends that chloride of tin is used merely as a mordant to fix these natural qualities; its use could be dispensed with, though not at present conveniently.

Mr. Allen said he thought they ought to distinguish between their present position and the position they might be in in the future. Dr. Dupré had raised the question how far they could obtain convictions under the Sale of Food and Drugs Acts in cases of mere colouring, provided that the colouring matter was harmless; but Mr. Cassal and the speaker had in recent papers foreshadowed the time when the present law might be amended; and the more instances they could bring before the notice of the authorities, and the more facts they had to go on, the stronger would be their position whenever the time came for fresh legislation. It was, of course, very desirable where possible to positively identify the nature of the colouring matter used, but that was by no means a necessity, any more than it was necessary for them to specify the exact nature of the fatty matters added to butter, or than it would be to specify the exact nature of hop substitutes supposing that any legislation were to be founded on the investigation of the recent committee. Mr. Allen said he certainly objected to have anything put into sugar for the sake of colouring it and making it pass as Demerara sugar. It was possible that such an addition might be dealt with under the Trades' Marks Act, but it appeared to him that the two facts taken together, namely, that it was not Demerara sugar, and that the colouring was put in for the purpose of deceiving, would make a good case to go before a magistrate.

Mr. B. E. R. Newlands said it would be a very extraordinary thing to find a sample of sugar that was not coloured. All sugars, such as loaf, granulated, crystals, and yellow crystals were coloured by artificial means, either with a blue or yellow colouring agent, and if the vendors of one kind of sugar were to be punished for introducing a harmless colouring matter, then those of all other kinds of sugar would be equally liable to prosecution.

In the West Indies yellow crystals were produced by the action of sulphuric acid or of stannous chloride, or, as at home, by the addition of a colouring matter. In the former cases, two or three gallons of the agent were added to a panful of sugar, and what Mr. Cassal would call a natural colour was produced. A good deal had been said as to palming off refined yellow crystals as Demerara, but if they referred to to-day's papers, they would find that, in many cases, the price of Demerara was lower than that of yellow crystals, the former selling at from 15s. to 16s. and the latter from 16s. 3d. to 17s. 3d. per cwt. It is, therefore, evident that the latter realise a higher price than the sugars they are supposed to imitate.

In Mauritius, Demerara, and the West Indian Islands, sugar, in addition to being coloured by sulphuric acid or stannous chloride, was coloured in exactly the same way as by refiners in this country, and therefore it would be impossible by any process of analysis to decide as to its place of origin.

What useful object could be attained by prosecuting grocers for selling yellow sugar, beyond providing work for the analyst? They might as well prosecute people for selling coloured sugar candy or Chelsea buns, or anything else coloured by artificial dyes. Dr. Dupré had suggested that the presence of artificial colouring matter in a sample of sugar might be ascertained by washing it with water. It was, however, extremely difficult to find a sample of sugar from which the colour could not be removed by water; he had been promised such a sample as a curiosity, but it had not yet arrived.

In turning out a standard article, such as yellow crystals, it was necessary to keep the colour perfectly uniform, and the refiner, therefore, had to add a minute amount of colouring matter, not exceeding one part in fifty thousand, for this purpose.

Manufacturers of sugar in the West Indies had often occasion to colour their products in the same way, and the sample of so-called "genuine Demerara sugar" handed round by Mr. Cassal, was thus artificially coloured. Whilst defending the sale of yellow crystals, he of course agreed that it was highly improper to describe them as "Demerara" unless they were actually produced in that colony.

Mr. Hehner said, that inasmuch as several aniline colours were of a decidedly poisonous nature, it could not be denied that the public analyst had the right and the *duty to concern* himself with the question of dyed sugar, and that he should know the exact nature of the colouring matters employed. He had no grounds for believing that injurious colours were used in this particular case, but just as a public analyst might not to know all details of technical and manufactory processes, so manufacturing chemists might possibly be ignorant of the physiological action of the colours which they added to food materials. As instances, Mr. Hehner mentioned Dinitro cresol (also known as Victoria yellow and under other names) and Dinitronaphthol (Martins yellow) which were both eminently poisonous.

Mr. Heron said he might say at once with regard to what Mr. Hehner said, that no colouring matters were used for colouring sugars which were of a poisonous nature; concerning the point brought forward by Mr. Stokes as to the use of chloride of tin in the West Indian plantations, such was not the case. As a matter of fact the chloride of tin was used there for the purpose of destroying the dark colouring matter present in the crudely prepared raw sugar and imparting to it a bright golden yellow colour so characteristic of Demerara sugar, and thereby making an inferior class of sugar appear of a better quality than it really was, and, as proof of his statement, he would ask any gentleman present, who was at all interested in this matter, to make a complete analysis of any West Indian sugar and compare it with an analysis of a home-refined sugar sold on the market as yellow crystal, and it would readily be seen that the latter was the purer of the two. He maintained that the colouring matter referred to by Mr. Cassal was not added to the sugar by refiners to give it a fictitious value, but merely to meet the public taste, which at present seemed to run in that direction, that this colouring matter was perfectly harmless to human beings and animals, and that the sugar sold as yellow crystal was in his opinion much purer and better in every way than any foreign sugar of a similar character at present on the market.

Dr. Dupre said that they as a scientific body should not accept the assertion of anyone, whoever he might be—let sugar manufacturers assert ever so much that the colouring matter they added was innocent—he should not pay the slightest regard to it unless they were prepared to state the nature of the colouring matter added, and then let him judge whether or not it was injurious to health. Did Mr. Newlands really mean to assert that all their sugar was artificially coloured? He was under the impression that some years ago the—what they call natural—colouring matter of sugar was a kind of caramel, and he should be exceeding astonished if Mr. Newlands told them that our old-fashioned sugar-candy was coloured with chloride of tin or sulphuric acid.

That colouring was what he called natural which remained in the sugar when treated with water. Mr. Newlands said there was no sugar now in the market the colour of which could not be washed out by water; that was to say that no sugar manufacturers really gave them unadulterated sugar.

Mr. B. Newlands said that the lower qualities of sugar-candy did contain the natural colouring matters of sugar, viz., caramel and dirt. The better qualities were produced from raw sugar which had been coloured by the agency of sulphuric acid, stannous chloride, etc.

Mr. Hehner said he would desire to protest against the often exploded doctrine that we should in any way be guided by the popular wish in food matters. The popular wish had been used by sophisticators for many years past as an excuse for every abomination perpetrated upon an ignorant public; no kind of adulteration was ever carried out without its being alleged to be in obedience to the popular wish. Years ago pickles and preserved peas were sold as green as the leaves on the tree, coloured with copper, and bloater-paste made red with brick dust; nowadays mustard and cocoa was mixed with flour and starch—all mixed by the obeying manufacturer to meet the popular wish. The public analyst, however, had to disregard the popular wish and raise the standard of purity of food, if necessary, *against* the popular wish. Was not even milk mixed with water because the people desired to have milk cheaper than it could possibly be produced by legitimate means? He hoped that public analysts would not allow themselves to be carried away by such phrases as "the popular wish."

Mr. Cassal, in reply, said he was very strongly of opinion that a public analyst was not bound to state the exact nature of an adulterant. He would go further, and say that it was always undesirable to enter into technical details of any kind before magistrates and others, who were utterly innocent of any scientific training, and therefore quite incapable of judging of the merits of the most elementary scientific questions.

Replying to Dr. Dupré, he stated that he had not as yet applied the spectroscope to attempt to identify the colouring matters, but that he proposed to do so. He desired to express his thanks as a public analyst to Mr. Newlands and to Mr. Heron for the opinions they had expressed and the admissions they had made. He was sure that the members of the Society generally would join with him in doing this, more especially those who occupied official positions as public analysts. At the same time he could not congratulate them upon the defence which they had endeavoured to make of the practice of colouring sugar with artificial dyes. He noted, with much satisfaction, that they did not agree with each other as fully as might have been expected, and that certain of the remarks which they had made were surprisingly contradictory. With reference to chloride of tin, for instance, the Society had been informed that it was used as a "mordant" to "fix" the "natural aroma, tint, and flavour of the sugar-cane," then that it was applied for the purpose of producing colour in colourless crystals; and, finally, "as a matter of fact," that it was used for destroying the dark colouring-matter of raw sugar. These statements appeared to require some revision by those who were interested in sugar manufacture, and he would leave the matter in their hands. It led him, however, to point out the necessity of distinguishing between sugars artificially coloured by producing a "natural" colour in the form of caramel, with such agents as chloride of tin or sulphuric acid, and dyeing with aniline dyes for a purpose which in all probability was very different. Mr. Newlands had told them that all sugars were coloured, and had referred to the use of blue for loaf sugar. It was hardly necessary to point out that this was an altogether different thing—it did not serve the purposes of deception. They had also been told about the prices, and he (Mr. Cassal) had been duly rated for meddling with a question which ought to be left to the superior ability of the "sugar expert," but he had also had the opportunity of consulting authorities even as great, perhaps, as those present, and the opinions they expressed were different upon both points.

It having been freely admitted that the dyed crystals were beet-root sugar, and that hardly anything else could be obtained on the market, he was glad to find Mr. Newlands agreeing with him that to sell "yellow crystals" as "Demerara" (which word implied *cane*-sugar) was highly improper unless they had been produced in that colony. In other words, beet-sugar should not be sold as cane-sugar, and anything which facilitated such a sale was therefore condemnable. In this connection he would remind members that of fifteen samples of sugar recently purchased as "Demerara" in Kensington, eleven consisted of dyed crystals.

What was the object of dyeing colourless sugar-crystals with an aniline dye or dyes? Mr. Newlands and his friends would not go so far as to deny that the dyes used were aniline derivatives of some kind. It was said that it was for the purpose of maintaining continuity of trade and of pleasing the public taste.

There was possibly some validity about the trade continuity argument, and he should be happy if he could bring himself to think that this was indeed the reason.

Butter and milk had been mentioned, and perhaps the continuity argument might apply in a limited sense to butter; he could not admit it for milk, and in reference to all food-products, colouring practices required to be sharply looked after. The object of most of them, if not all, was to produce a false impression on the mind of the customer; and, as had been pointed out, they were fraught with danger. The only satisfactory method was to treat the matter rigidly, and to make it compulsory upon all vendors of food containing extraneous products to state what they had done. The "public taste" excuse for adulteration had been fully dealt with by Mr. Hehner; it was a very ancient excuse, and nothing more need be said about it.

They had been told that all the colours used, or likely to be used, were absolutely harmless in any amount. That was a somewhat bold statement, and he (Mr. Cassal) fully agreed with what Dr. Dupré had said about it. Something more was wanted than the mere statement of a particular person or firm that what they in their wisdom chose to put into food was "perfectly harmless" on the strength of some nebulous experiments made by somebody—they knew not whom—somewhere.

The samples moistened with acid which he had passed round were intended, as he had said, to show the difference between a sugar which would give the peculiar pink colour with hydrocholoric acid, and one which would not. He thought that he had made it sufficiently plain in his paper that he relied upon the removal of the dyes by suitable solvents, and their purification if necessary; and not simply upon the incident that some of these dyes could be detected at once by the addition of a mineral acid.

The attempt alluded to in the paper to prevent the maintenance of the definition which could at present be given to Demerara sugar by stating that cane-sugar produced in Demerara was there coloured with aniline dyes and then exported was certainly ingenious, and reflected great credit upon whoever had evolved the idea; but it was singular that those who believed in this theory should admit that nearly all, if not all, the dyed sugar on the market was beet. Even if it was admitted that dyes were applied to cane-sugar in Demerara, he contended that the case was not affected. It was simply adulteration in Demerara instead of in England. By Demerara sugar was understood cane-sugar, and cane-sugar containing only such colouring matter as was natural to sugar.

He thought that the Merchandise Marks Act could certainly be applied to the case, and if it were so applied some of the vendors of "crystals" or "Demerara" might be disagreeably surprised. The penalties were not likely to be of the trumpery kind to which they were accustomed under the present Adulteration Acts. He was much gratified to find that the general consensus of opinion in the meeting was in favour of the views which he had put forward.

(*Conclusion of the Society's Proceedings.*)

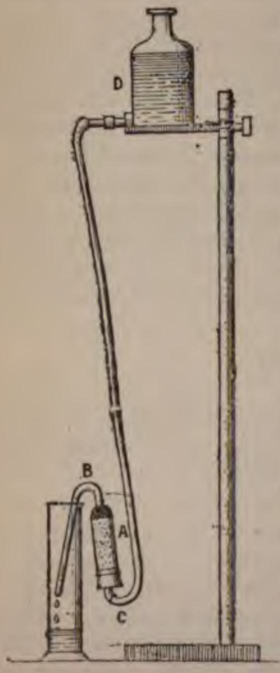
METHODS AND APPARATUS IN USE AT THE LABORATORY OF THE BOURSE DE COMMERCE AT PARIS FOR THE ANALYSIS OF CERTAIN COMMERCIAL ORGANIC PRODUCTS.

By Dr. Muter.

(*Continued from page* 89.)

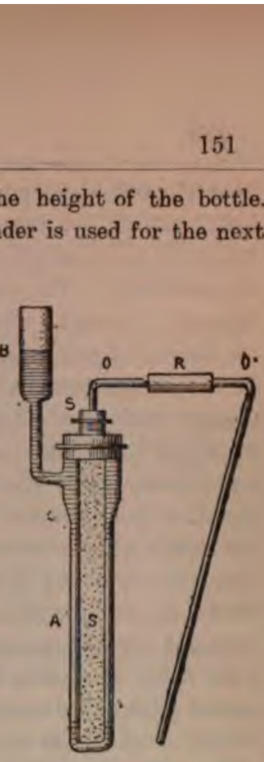
The process adopted for the valuation of all sorts of tanning materials is the well-known one which consists in ascertaining the amount of absorption taking place from a solution of the tanning material when brought into contact with prepared hide or with Seimand's ossein, the result being reported under the name of "available tannin." The arrangements for simplifying the process and rendering it sufficiently rapid to admit of its being done at the small commercial fees charged at the laboratory are (like most of M. Jean's arrangements) ingenious and effective. Before describing them in detail, it will be interesting to state that, disagreeing with the usual method of taking the specific gravity of the liquid before and after the maceration with the hide, M. Jean, for good and sufficient reasons, which he explained to us, considers it to be necessary to take the actual residue left on evaporation. The following is a short description of the process as actually in daily use :—

1. *Extraction of the Crude Material and Preparation of the Solution for Analysis.*—The bark is ground in the mill, or dry extracts are pounded in the mortar, so that a powder is obtained which will pass through an ordinary "25" sieve. The quantity to be taken for analysis is, of oak bark, 25 to 30 grams.; of stronger things (such as chestnut wood), 20 to 25 grams.; while, of very rich extracts, it may only be needful to weigh 10 or 15 grams. The quantity to be weighed is always to be judged by remembering that the solution, when actually made up to 250 c.c., must contain as nearly as possible 1 per cent. of "assimilable tannin." The sample having been weighed, is now extracted by continuous percolation in a special apparatus. This consists of a cone of wire-gauze, which is tinned or nickel-plated, and into which the weighed quantity of the sample is put. This gauze receptacle is then introduced into an outer tube, connected to an upright condenser, and to the foot of the tube is attached, by means of a cork, a flask of 150 c.c. capacity, having 100 c.c. of water placed therein. The water in the flask being boiled, continuous extraction takes place, which entirely exhausts any material within the space of one hour. In the Paris Laboratory, where a large number of samples are being constantly dealt with, there are a series of condensing tubes, all passing through one large outer case of zinc, on which cold water streams, so that a whole row of extractions may be done at the same time. When the condensed liquid drops back into the flask perfectly colourless, the extraction is at an end, and the liquid is rinsed from the flask into a measure, and made up to 250 c.c. As a rule, this liquid is not perfectly clear, and seeing that it must be so before applying the powdered hide, we have next to proceed to :—

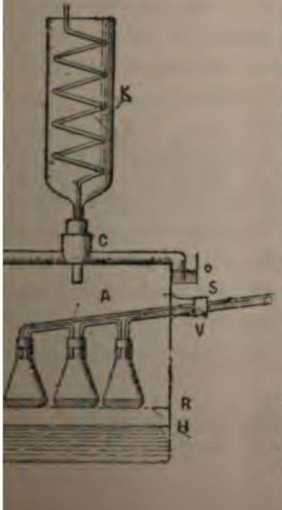


2. *The Filtration of the Liquid.*—This is done by means of the apparatus shown in figure 2. A is a short wide tube, having another tube (B) fused into its lower end, B being bent in the form of a syphon. To prepare A for use, a piece of wire-gauze covered by flannel is first dropped in, and then that is followed by a pulp made by macerating filter-paper in distilled water for a few minutes. When A is thus filled, the operator's lips are applied to the end of B, so as to suck out the excess of water and cause the pulp to mat together. The mouth of A is then closed by an india-rubber cork, through which passes a bent tube (C), which is in turn attached by means of an india-rubber tube, about 2 ft. long, to the bottom outlet of the bottle D, this bottle being placed on a stand as shown. The liquid to be filtered is poured into the bottle D, and the tube B is hooked over a receiving jar. The liquid now begins to

and the rapidity of filtration is regulated by altering the height of the bottle. irst 20 c.c. that pass through are rejected, and the remainder is used for the next f the process.



. *Extraction of the Tannin by the Powdered Hide.*—is done in the apparatus shown in figure 3, and known "Tannimetre Jean." A is a glass tube, closed at the n, and having a cylindrical funnel and bent tube (B) fused ts upper part. Inside A an inner tube (S) is fixed by of an india-rubber ring. The tube S is in turn closed india-rubber stopper, through which passes the bent O. This tube is joined to a corresponding portion by of a long cork (R), so that, when O is turned down, hole forms a syphon with the inner tube S. By g O in R we can establish or stop the syphon action asure. To prepare the apparatus for use, we close the f S with a piece of wire-gauze covered with flannel, and en pack it with the powdered hide, and place it in posi- nside A as shown. The liquid to be exhausted is intro- into the funnel B *very slowly*, so that it is gradually l up into S by capillary action, the tube O being at the same time kept l up to prevent any action of the syphon. When the liquid in the two A and S have both reached the level of the point where the funnel is fused into A, some more of the liquid is poured into the funnel B, to raise the level of liquid just short of the bend in O, and the whole d to stand for one hour. At the expiration of this time, the funnel is nearly filled he liquid, and the syphon having been turned down, filtration is established and ued until 40 c.c. have dropped from the longer limb of O. If the apparatus orked properly, the filtrate should be limpid, almost colourless, and should not give a precipitate with gelatine. The powder used is the article produced at the Imperial Tannery in Vienna, and can be purchased with the apparatus from Demichel at Paris. When a new lot of powder is purchased, it is necessary to make a blank experiment by charging the "tannimetre" therewith, and using distilled water instead of a tannin solution, so as to obtain an extract. By now evaporating 25 c.c of this liquid and weighing the residue, we obtain the check for the particular lot of hide powder. Such check is, however, very constant, and is ·016 grams. for the ordinary powder containing 15·15 per cent. of moisture.



4. *Obtaining and Weighing the Residue.*—25 c.c. of the solution that has not passed through the tannimetre, and another 25 c.c. of that which has so passed, are each evaporated in a weighed flat-bottomed glass capsule at

212° F., and the dry residues are weighed. The difference between the two weights, less the check, gives the amount of "assimilable tannin," and this result, multiplied by 10, gives the quantity in the weight taken for analysis, and, lastly, this is calculated to percentage. The apparatus actually employed for drying the residues is shown in figure 4. It works with great rapidity, and is manifestly adapted to the obtaining of the residue of any fluid of which a number of samples are to be done at the same time. It is a copper bath (A) with amovable top (C), through which passes the end of an upright condenser. A trough containing sand goes all round the top of the bath, so that, when the lid is put on, it fits steam-tight. Water is put in up to the level (H), and immediately above there is a sheet of wire-gauze (R) for the articles to stand on. The liquid, of which the residue is to be obtained, is placed in a tarred, flat-bottomed glass flask, the base of which is about 2·5 in. in diameter. When a series of flasks have been charged they are closed by india-rubber corks, through which pass the arrangement of tubes shown in the drawing, such tubes projecting through a side tubulure (S) specially arranged for the purpose. The water in the bath having been set to boil, one of the glass tubes projecting through S is attached to an empty flask, and this is in turn connected with a water vacuum pump. The whole of the flasks being immersed in vapour at 212°, and all the vapour being drawn off as fast as it forms, the evaporation proceeds with extreme rapidity. When no more liquid condenses at the extremity of the exhausting tube, the inlet tube is attached to an air-drying arrangement, so that a current of perfectly dry air is caused to pass over the residues, and the absolute desiccation is rapidly achieved.

ON WAX ANALYSIS.

By M. H. Rottger.*

The method most commonly employed for the analysis of beeswax is based upon Köttstorfer's process for the analysis of fats by two successive saponifications, as follows:—Five grams. of the wax are warmed with 20 c.c. of alcohol (of 95°) until they fuse, and a few drops of alcoholic solution of phenol-phtalein having been added, the whole is shaken and titrated, while still warm, with semi-normal solution of potassium hydrate, delivered from a burette divided into ·5 c.c. From the result of this experiment the quantity of potash, in millegrammes, necessary to saturate the cerotic acid contained in one gramme of the wax is calculated and is put down as the first number. Twenty c.c. more of the potash solution are then run in, and, the flask having been connected with an upright condenser, the whole is heated until entire saponification has taken place. When this is attained, the flask is brought under a burette containing semi-normal hydrochloric acid, and titrated until the colour is just discharged. The number of c.c of hydrochloric acid so used deducted from 20 gives the c.c. of the potash solution used up in the act of saponifying the myricyl palmitate; from this is calculated the amount of potash in millegrammes necessary for one gramme of the wax, and the figure so obtained is called the second number. Finally, the first and second numbers are added together to obtain the third number.

Numerous researches have shown that in pure yellow beeswax the first number is mmes (or between 19 and 21); that the second number is 75 millegrammes (or 76); while the third number is 95 (or from 92 to 97). The relation between nd second numbers in genuine wax is as 1 to 3·7 on the average, but may en 1 to 3·6 and 1 to 3·8, and the third number must not be departed from in e sample.

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* *Monit. Scient.*

tly, G. Buchner has analysed a series of samples, and has given the following nding to show that this theory breaks down when we have to deal with wax been bleached by chemical means. The following are Buchner's results:—

	1st number.	2nd number.	3rd number.	Relation.
ax	20·2	73·8	94·8	1·3·65
y-bleached wax	19·91	75·0	94·9	3·76
	19·80	74·9	94·7	3·78
lly-bleached wax	21·97	76·4	98·37	3·47
	22·00	75·9	97·9	3·45
.. ..	24·2	74·8	99·0	3·09
	23·8	74·5	98·3	3·13
	23·9	74·5	98·4	3·11

these figures Buchner concludes that the standard relation in chemically- mples must be taken as 1 to 3·2.

t the accuracy of these results some experiments have been done in the labo- elfenberg. Two varieties of genuine yellow wax were started with, and each n divided into two parts, the one was bleached by the charcoal process and by potassium permanganate. The results obtained from these undoubtedly es contradicted those of Buchner, and fully confirmed the original view. They lows:—

	Density.	1st number.	2nd number.	3rd number.	Relation.
.. .. ..	0 966	19·6	74·0	93·6	3·77
ed by charcoal ..	0·963	20·1	74·0	94·1	3·68
„ $KMnO_4$ ..	0·963	18·1	72·8	90·9	4·00
(second sample) ..	0·964	20·1	74·8	94·9	3·72
ed by charcoal ..	0·963	19·6	74·0	93·6	3·77
„ $KMnO_4$ ..	0·964	19·1	74·0	93·1	3·87

gures thus obtained showed that the process of bleaching does not increase gravity of wax, as some observers have pretended, and also that the three ic numbers are no larger, and are comprised within the same limits as those yellow wax. I have lately also had occasion to analyse white wax chemically- the acid process, and the following are the results I obtain:—

Specific gravity.	1st number.	2nd number.	3rd number.	Relation.
0·966	20·2	76·7	96·9	3·79

numbers also contradict Buchner's hypothesis, and I am afraid that the so- white wax with which he was supplied was scarcely what it should have is necessary to be very cautious accepting commercial descriptions, as is he following analysis of two samples of white wax, sold as of a superior which were evidently very far from meriting the epithet:—

1st number	29·7	31·46
2nd number	87·5	76·66
3rd number	117·2	99·1
Relation	2·9	2·14

nclusion, I would urge that there is no reason for adopting any different r judging the purity of white wax to those employed in the examination of

ESTIMATION OF ALKALIES AND ACIDS BY MEANS OF IODINE.

By Max Gröger.*

The principle of this process is the action of sulphuric acid on a mixture of potassium iodide and potassium iodate, which is represented by the equation—

$$KIO_3 + 5KI + 3H_2SO_4 = 3K_2SO_4 + 3H_2O + I_6.$$

Alkaline solutions are first mixed with excess of sulphuric acid and then with a solution of potassic iodate and iodide (which I will now simply call the iodine solution); acids are at once mixed with the iodine solution, and in both cases the liberated iodine is determined. What we want is a $\frac{n}{10}$ solution of sodium thiosulphate, $\frac{n}{10}$ sulphuric acid, and a perfectly neutral solution of the iodine.

The thiosulphate: 25 grammes of the crystallised salt are dissolved in a litre of water. Although the exact strength of the solution can be determined by a known weight of pure iodine, I prefer to operate as follows: About ·15 grammes of pure potassium iodate is accurately weighed, dissolved in a little water, mixed with about 1 gramme of pure potassium iodide and acidified with hydrochloric acid. The thiosulphate is then run in until the solution is decolorised, starch solution being finally used as indicator.

The sulphuric acid: 5 grammes of concentrated sulphuric acid are diluted up to a litre. A measured quantity is then mixed with excess of the neutral iodine solution and the liberated iodine determined as usual.

The iodine solution: By dissolving iodine in potash ley a solution is obtained which contains the proper proportion of iodide and iodate—

$$6KHO + I_6 = 5KI + KIO_3 + 3H_2O,$$

but one does not succeed in getting a perfectly neutral solution in this manner. If to the warm ley (free from carbonic acid) iodine is added as long as the solution remains colourless, it remains strongly alkaline. If, on the other hand, iodine is added until the fluid is dark brown, and the whole is then thoroughly boiled, the fluid certainly becomes neutral, but always contains some free iodine. It is therefore the best plan to prepare perfectly neutral solutions of potassium iodide, and ditto iodate, and to mix them in the proper proportions. In airtight bottles the mixture may be kept unchanged, but in contact with air the carbonic acid gradually liberates iodine, and the mixture becomes yellowish and also faintly alkaline. It is therefore better to keep both solutions separately, and only to mix just before use. The solutions used in my experiments respectively contained 4 grammes of potassium iodide and 24 grammes of iodate in 100 c.c. When equal volumes were mixed a half normal solution was obtained. It is not necessary to know its exact strength.

Estimation of Caustic Alkalies. 50 c.c. of the watery solution (about $\frac{n}{10}$ strength) were mixed with excess of $\frac{n}{10}$ sulphuric acid, then with 5 c.c. of the iodine solution, and titrated as usual with thiosulphate. The difference in c.c. of acid and thiosulphate equals the amount of acid required to neutralise the alkali. I also estimated the alkali with acid, using litmus or phenol-phthalein as indicator.

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* *Zeitschr. f. Angew Chemie*, No. 12, 1890.

C.C.S OF ACID REQUIRED.

Solution of.	Iodine process.	Litmus.	Phenol-phthalein.
KHO	39·85	39·80	39·85
"	27·05	27·03	27·05
NaHO	42·40	42·42	42·40
"	35·35	35·30	35·30
NH_3	31·55	31·50	—
"	48·20	48·25	—

Estimation of Alkaline Carbonates.—First of all I investigated the action of free bonic acid on the iodine solution. A litre of distilled water, saturated with carbonic d, evolved by ignition of sodium bicarbonate, showed on titrating with baryta water, 89 gramme of carbonic anhydride. This carbonic acid solution was now mixed with c.c. of the iodine solution, and from time to time 50 c.c. were titrated with the iosulphate, with the following results:—

Directly after mixing	·18 c.c. $\frac{n}{10}$ thiosulphate.
" 5 minutes	·24 " "
" 10 "	·27 " "
" 15 "	·30 " "
" 30 "	·54 " "
" 60 "	·72 " "
" 2 hours	·90 " "
" 3 "	·96 " "
" 24 "	1·68 " "
" 48 "	1·86 " "
" 72 "	1·92 " "
" 120 "	1·92 " "

The carbonic acid therefore decomposes the iodine solution quickly at first, and adually slower and slower, until after three days the action completely ceases. The composition is very incomplete. Then according to the equation

$$KIO_3+5KI+3CO_2=3K_2CO_3+I_6$$

c.c. of this carbonic acid solution ought to have used 31·58 c.c. thiosulphate.

If the solution of an alkaline carbonate is mixed in the cold with an excess of $\frac{n}{10}$ id, mixed with iodine solution and titrated with thiosulphate, the liquid after remain-g colourless for a moment, rapidly gets blue again. If the thiosulphate is again added drop at a time, the fluid will get each time blue again, and this keeps on for about an our. If, however, so much thiosulphate is added that the fluid remains colourless for a w seconds, the results do not materially differ from those obtained by the usual processes.

50 c.c. of solution of alkaline carbonate were mixed in the cold with 50 c.c. of $\frac{n}{10}$ lphuric acid, then after addition of 5 c.c. iodine solution, titrated with thiosulphate ntil the liquid was colourless. After standing for thirty minutes the blue colour was nce more destroyed by more of the reagent. Another 50 c.c. were mixed with 50 c.c. f acid, boiled to expel carbonic acid, and titrated back with $\frac{n}{10}$ potash.

C.C.S OF ACID CONSUMED.

Solution of	Iodine process. Direct.	Iodine process. After 30 minutes.	Phenol-phthalein.	Litmus.
K_2CO_3	43·82 ..	.. 43·65	43·91	43·90
Na_2CO_3	28·06 ..	.. 27·80	28·15	28·15
Am_2CO_3	29·54 ..	.. 29·49	—	29·56
$KHCO_3$	48·08 ..	.. 47·58	48·15	48·17
$NaHCO_3$	46·87 ..	.. 46·35	46·96	46·99
$AmHCO_3$	32·90 ..	.. 32·46	—	33·00

The influence of the carbonic acid is plainly visible from this table. The results obtained on titrating immediately after mixing are practically the same as those obtained by the litmus process, but those obtained after allowing the mixture to stand for thirty minutes are too low, more particularly in the case of the bicarbonates.

If, however, the iodide solution is added after all carbonic acid has been expelled by boiling and the fluid got cold again, perfectly concordant results may be obtained. For delicate analyses this course is therefore to be recommended.

Estimation of Alkaline Sulphides.—Hydrogen sulphide is completely decomposed by iodine solution :

$$KIO_3 + 5KI + 3H_2S = 6KI + 3H_2O + S_3.$$

If, therefore, solutions of the alkaline sulphides or hydrogen sulphides are mixed with a known excess of $\frac{n}{10}$ acid, and then with a sufficiency of iodine solution, the liberated iodine may be determined by means of thiosulphate, but even when a slight excess of the latter has been added the liquid still has a dirty blueish colour, because the separated sulphur encloses some of the iodide of starch. I made experiments with solutions of caustic alkalies before and after saturation with hydrogen sulphide, with the following results :—

C.C.s OF ACID USED.

50 c.c. solution of	Direct.	After saturating with H_2S.
KHO	30·04	29 36
,,	42·30	41·90
NaHO	27·59	27·03
,,	44·25	43·85

If, however, the hydrogen sulphide is first expelled by boiling, the results are satisfactory, as will be seen from the following series of experiments. The results were checked in the usual way.

C.C.s OF ACID CONSUMED.

Solution of	Iodine process.	Litmus.	Phenol-phthalein.
K_2S	25·75	25·70	—
Na_2S	32·04	32·06	32·04
Am_2S	39·82	39·79	—
KHS	48·31	—	48 25
NaHS	19·57	--	19·60
AmHS	45·55	45·55	—
K_2S_5	28·20	28·06	—
Na_2S_5	32·18	32·15	32·18
Am_2S_5	17·05	17·00	—

The sulphur, which separates on the addition of acids to polysulphides, does not interfere with the titration.

If, therefore, a very accurate analysis is wanted, it is best to expel the hydrogen sulphide by boiling, but if its amount is but very small this is not necessary. The fading of the blue colour is, as a rule, easily to observe, even in somewhat dark-coloured liquids, such as wood ash lye, soap lyes, ammoniacal liquid from gasworks, etc., which makes the iodine process preferable to the titrating with litmus or methylorange. Although this last indicator has got much in favour lately, I must confess I have some difficulty in noticing the end reaction, if the fluid under examination is somewhat yellowish or brownish.

Estimation of Acids.—50 c.c. of acid solution were added to 10 c.c. of iodine solution, and the liquid at once titrated with thiosulphate. A check was made by $\frac{n}{10}$ potash:

	Used.	
Solution of	C.C. of thiosulphate.	C.C. of potash.
H_2SO_4	41·90	41·80
"	17·55	17·55
HCl	28·14	28·20
"	35·70	35·70
HNO_3	42·87	42·92
"	15·05	15·10

Organic acids act but very slowly in the cold, requiring some 24 hours. With acetic acid the action was not even then complete, 50 c.c. of a weak solution requiring 38·55 c.c. instead of 40·3 c.c. thiosulphate. As, however, heating very much promotes the action, I fancy good results may be obtained by first distilling off the liberated iodine. If successful it will be an excellent process for determining the acidity of wines, beers, and other dark-coloured liquids.

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Assay of Gum Arabic and Gum Senegal. By Liebermann. (*Chem. Zeit.*, No. 41, 1890).—1. Gum arabic forms round or angular, colourless, yellowish or brownish lumps, which strongly refract the light, and look as if they possessed a crystalline structure. Owing to the disturbed state of trade in the Soudan, much of the gum arabic nowadays imported is partly, or even totally, composed of gum senegal.

2. Gum senegal forms either colourless or yellowish lumps, somewhat whitish on the surface (resembling corroded glass), the interior of which is, however, clear and lustrous. The lumps are generally longish, straight or bent, vermicular or cylindric. Sometimes they look as if small lumps have deposited round a larger one. They have to a certain extent the shape of mulberries. If, therefore, the sample is not in powder or too small lumps the very appearance will tell the fraud.

3. Both varieties are completely soluble in water; there only remains small particles of wood, which, in samples of gum arabic, are generally reddish, but blackish in the gum senegal. These woody particles are found even in the superior kinds of the gum. Other gums, like cherry gum, are only partly soluble in water. There remains a jelly which only dissolves on prolonged boiling.

4. The watery solution of both gums gives with potash-ley and a few drops of solution of copper sulphate blueish precipitates; but with gum arabic the precipitate is more abundant, sticks together, and rises to the surface of the fluid. The precipitates are not dissolved on heating and do not reduce the copper.

5. Dextrine solution also gives a blue precipitate, but this dissolves completely on warming to a clear dark-blue fluid. On prolonged boiling the copper gets completely reduced.

6. Heated for a long time with dilute potash, gum arabic or dextrine turn amber-yellow, whilst gum senegal scarcely colours at all.

7. Mixtures of the two gums behave towards potash and copper sulphate like pure gum senegal, but on boiling with potash alone the mixture turns amber-yellow.

8. Mixtures of gum arabic and dextrine behave towards potash and copper sulphate like pure gum, but on long boiling reduction takes place if at least the amount of dextrine is not too small.

9. To detect *small* quantities of dextrine the liquid must, after a slight warming, be filtered before boiling.

10. In similar manner the separation must be performed when both kinds of gum are present, as well as dextrine. The cupric precipitate containing both gums is washed with distilled water, dissolved in a little dilute hydrochloric acid, and mixed with a large excess of spirit. After standing for a day, the transparent deposit is dried, then dissolved in hot water, and tested according to 4 and 6.

The assay of gum arabic may also be prepared according to the following scheme:—

(*A*). The appearance of the sample when not in powder. (See 1 and 2.)

(*B*). Try the solubility of the powdered sample in warm water. (See 3.) If the sample is but partially soluble, and leaves a jelly-like mass, there is no doubt cherry gum. If practically soluble, the solution is mixed with excess of potash and a little copper sulphate, gently heated and filtered:

(*a*). The filtrate is treated for dextrine according to 9.

(*b*). Precipitate is treated as described in 10. The deposit will either agglomerate and float, or remain suspended in the fluid. In the first case there is gum arabic, and the orginal fluid will turn amber-yellow with potash. In the second case if there is no colour got with potash, there is only gum senegal.

It has been said gum senegal is more hygroscopic than gum arabic. To make sure the author dried both specimens at 105° C., and then exposed them for twenty-four hours to moist air, when gum arabic was found to be even a trifle more hygroscopical than the gum senegal. L. DE K.

A NEW INDICATOR: (A) NAPHTOL-BENZEIN. R. LALOZIECKI. (*Chem. Zeit.*, No. 37, 1890).—The author prepares this substance by mixing two molecular quantities of (a) naphtol with one benzol trichloride, after adding some benzol to mitigate the action. After standing for twenty-four hours the whole is heated up to 40° C., and the benzol, with the excess of the trichloride, driven off in a current of steam. The product is then purified by repeated solution in soda lye, and precipitation with hydrochloric acid. In volumetric analyses ten or twenty drops of its 1 per cent. spirituous solution is added to the fluid until the acid changes the liquid from green to orange, or the alkali from orange to green. In the latter case it is, however, preferable to add a slight excess of alkali, and titrating back with acid.

The change is plainly visible, even with artificial light. L. DE K.

A DELICATE REAGENT FOR SOME ESSENTIAL OILS. A. IHL. (*Chem. Zeit.*, No. 27, 1890).—Pyrol acts very characteristically on the incrustating material of wood. The author thought of trying its action on ethereal oils, and obtained the following results:—

innamon oil: A very weak alcoholic solution of this oil gives with dilute alcoholic lution of pyrol, mixed with hydrochloric acid, first a yellowish-red colour, which soon rns dark red, and finally deposits a dark-coloured substance. This test is a delicate e for both pyrol and the oil. Oil of cloves: This, when mixed with a dilute alcoholic lution of pyrol and some hydrochloric acid, gives a splendid carmine colour. The same curs with pimento oil. Oil of sassafras gives a splendid rose-red colour. Spearmint l gives but a faint reddish colouration, and aniseed oil only a yellowish colour. The thor thinks these oils are closely related to the woody substance. L. DE K.

SIMPLE METHOD FOR THE ESTIMATION OF EXTRACTS IN WINES. ED. LASZLO. *'hem. Zeit.*, No. 27, 1890).—It has several times been remarked how important it ould be to everywhere determine the extract of wine by the same process. Now, this done in nearly every country by a different method, causing different results, and con- quently great dissatisfaction. In Austria, and also in Germany, the direct process is vogue, although, as has often been remarked, the results do not really represent the ue amount of extract, but simply the percentage happening to be left after drying for ro hours and a half. The author gives a few instances of the unreliability of this ethod. Just to mention one: Two wines, having exactly the same specific gravity and e same percentage of alcohol, still showed a difference of ·24 per cent. in extract. These ines were analysed by the same chemist. Even the lowest result was still ·21 per cent. excess of the calculated quantity. A process which gives such contradicting results nnot really be tolerated.

As regards the indirect process, the author refers to the pamphlet of B. Haes, but so calls attention to Pohl's thermo-areometric test. The extract gravity is calculated om the percentage of alcohol, and the present gravity of the sample (extract avity = present gravity + [1 − alcoholic gravity]). Pohl used for his alcohol estima- ons his vaporimeter, which gave varying results, so his extracts were therefore faulty; d, although we now possess Malligand's vaporimeter, the process seems to have got ite out of use. The author now uses a very accurate hydrometer, which enables him read off ·1 per cent. of alcohol. It shows percentage of alcohol by volume, and must used at 15° C. If used for wine it will, of course, show the less alcohol, as there is ore extract. The difference between real and apparent alcohol may serve, of course, as basis of calculation of the extract. $E = f\ (a - s)$, in which equation E represents the nount of extract in 100 A; A the actual amount of alcohol by volume, determined, say, distillation; S the apparent amount of alcohol found by the hydrometer; and F, a rrection which the author proposes to fix as ·32.

If the sample should be heavier than water, the author uses a Billings' saccharo- eter (hydrometer), but calculates its percentages by weight to ditto by volume. In alysing sweet wines, he therefore multiplies the alcohol by volume with ·32, and adds e saccharometer degrees, calculated to volume. The test analyses are very satisfactory. a sample containing 12·0 per cent. of alcohol and 2·5 per cent. of extract, the latter as found to be 2·52 by the author's process. The apparent percentage of alcohol as 4·1. L. DE K.

DETECTION OF STEARIC ACID AND PARAFFIN IN WAX. H. RÖTTGER. (*Chem. Zeit.* o. 37, 1890).—The author criticises the various methods from time to time proposed. egnard's process: The sample is boiled with water and then allowed to cool. If stearic id is present, the watery fluid will slightly redden blue litmus paper. This chemist, wever, confesses that the acidity may, in reality, be caused by traces of sulphuric acid used in bleaching the wax. He also proposes to boil the sample with lime-water in a flask attached to a reflux condenser, when, if stearic acid is present, a flocculent deposit of calcium stearate forms. Geith also uses the lime-water test, but does not mention anything about the precipitate; only says the lime-water loses its alkalinity. The author's experience of this process is that a precipitate forms at first, but disappears on boiling. It depends on the strength of the lime-water or on the amount of stearic acid whether the watery fluid will present an alkaline, or even an acid, reaction. Hayer shakes a chloroform solution of the sample with twelve volumes of lime-water very suddenly. If stearic acid is present, there forms a lime soap in dendritic and granular ramifications, whilst the chloroformic fluid neatly separates as a white emulsion. The author cannot recommend this modified process. Fehling says: As pure wax contains cerotic acid, which, in its behaviour to caustic and carbonated alkalies, resembles stearic acid, the test with lime-water cannot succeed if the adulteration is below 10 per cent. He recommends the following plan:—The sample is boiled for five minutes with twenty times its weight of alcohol, and then allowed to cool. After standing for a few hours, the liquid is filtered off and mixed with water. If the sample was pure there will be but a faint opalescence, as cerotic acid, although soluble in boiling alcohol, almost completely separates out on cooling; but if as little as 1 per cent. of stearic acid is present it will cause a decided flocculent precipitate. The author finds this a most elegant and reliable process. Benedict boils the sample with solution of sodium carbonate, when, if stearic acid is present, the alkaline fluid will, on cooling, gelatinise. The author finds this process to be less safe than the previous one, especially if the adulteration is rather small.

Detection of Paraffin and Ceresin. Landolt's Process.—Fuming sulphuric acid completely destroys wax; paraffin is not attacked. The sample is heated in a porcelain dish with this acid in excess, when, after some time, the paraffin will float on the top of the fluid. This not very elegant and very uncertain process is not of much use nowadays, as the kind of paraffin now employed for wax adulteration is also destroyed by the acid. Wagner calculates the amount of paraffin from the specific gravity. He argues: Genuine wax has a constant specific gravity, whilst the various kinds of paraffin do also not greatly differ in gravity from each other. He gives the following table:—

Wax.	Paraffin.	Specific gravity.
0	100	871
25	75	893
50	50	920
75	25	942
80	20	948
100	0	969

Pure wax (free from paraffin) ought to sink in spirits of wine of 961 specific gravity. Donath, after making sure that no other adulterations are present, also calculates the amount of paraffin from the specific gravity. If the gravity of the sample should fall below 960, he takes the presence of paraffin for granted. Buchner made several determinations of the specific gravity of various kinds of ceresin, and found it to vary from 858 to 901. Wax containing ceresin still floats on spirits of wine of 950 specific gravity. For the chemical detection, he boils the wax with alcoholic potash (1-3), when paraffin and ceresin, not being saponifiable, will float on the surface. Hager heats 2 grams. of the wax with 5 c.c. solution of common soda, shakes well, and adds 6 c.c. of benzol, when an emulsion forms. After standing for an hour in water of 50° C., two layers form. If the wax is pure, the benzol will, after cooling, be almost clear; but in the presence of paraffin it will be thickish and turbid. Sedna puts the wax shavings with twenty times their amount of ether for half a day. Wax scarcely dissolves, but the paraffin does. Of the ethereal residue five-sixths is called paraffin. L. DE K.

THE ANALYST.

SEPTEMBER, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

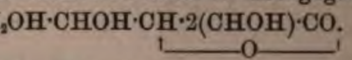
ON SOME RECENT ADVANCES IN OUR KNOWLEDGE OF THE SUGAR GROUP.

BY WALTER J. SYKES, M.D., D.P.H., F.C.S., ETC.

(*Read at Meeting, June,* 1890.)

THE synthesis of the sugar group, a process so intimately connected as it is with the physiology of plant life, has naturally formed an attractive subject of investigation amongst chemists. Numerous attempts have been made from time to time to form these bodies synthetically, but hitherto only a small measure of success has attended the efforts made in this direction. Quite recently, Dr. Emil Fischer has solved this problem in a most brilliant and satisfactory manner, since he has not only synthesised from the elements glucose and the whole mannitol group of sugars, but has also discovered new members of this group, of the existence of which we previously knew nothing. His labours throw considerable light on the molecular constitution of the individual members of the group as well. My paper of this evening is a brief *resume* of his important work in this direction.

The ordinary hexa-hydric alcohol mannitol $CH_2OH\cdot 4(CHOH)\cdot CH_2OH$, obtained by extraction with alcohol from ordinary manna, yields upon limited oxidation with HNO_3 a body very similar in its properties to glucose. It reduces Fehling, ferments in contact with beer yeast, and rotates the polarised ray to the right, though not to such a wide angle as glucose. This body mannose is the aldehyde of mannitol, and has the following constitution :—$CH_2OH\cdot 4(CHOH)\cdot COH$. Upon further oxidation with Br it yields the corresponding mannonic acid $CH_2OH\cdot 4(CHOH)\cdot COOH$. If a solution of this acid be evaporated to a syrupy consistence, a molecule of water is disengaged, and the lactone or anhydride of the acid produced



$$CH_2OH\cdot CHOH\cdot \underbrace{CH\cdot 2(CHOH)\cdot CO}_{O}.$$

This body crystallises in stellate groups of colourless needles, melts at 149 to 153° C., and has a specific rotary power of $[\alpha]_D + 53{\cdot}81$. Its solution has a neutral reaction; it, however, readily decomposes carbonates, and forms the corresponding mannonates.

Killiani some years since obtained by boiling certain gums with dilute H_2SO_4 a penta-hydric aldehyde (sugar) to which he gave the name arabinose. This sugar upon treatment with HCN yields the cyan-hydrin, and this in its turn upon treatment with $Ba(HO)_2$ yields the acid of the alcohol of the next degree higher in the series. This he called arabinose-carboxylic acid; it has the same composition as the mannonic acid mentioned above. On evaporation the arabinose-carboxylic acid yields its lactone.

In this method of Killiani's we have obviously a method of passing up the alcohol series, from lower to higher, and to which I shall have to revert further on.

The lactones of the mannonic and arabinose-carboxylic acids have obviously the same structural formulæ, and are so similar in other respects that they must have been considered identical, had not their optical properties differed. Mannonic acid lactone polarises $[\alpha]_D + 53{\cdot}81$, the other lactone $-54{\cdot}8$; in other words, making allowance for experimental error, these two bodies possess equal and opposite optical activities, and may, therefore, be looked upon as two optically opposed isomerides like the dextro and lævo-tartaric acids. This is the first instance observed of the occurrence of such a relation in the sugar group. If such be the case, it is obvious that in a solution of equal quantities of the optically opposed lactones, combination ought to take place, and an inactive lactone be produced, as in the parallel case of the two tartaric acids, which under similar conditions unite and form racemic acid. The experiment was tried, and found perfectly successful; an inactive lactone was produced which could only be split up again into its optically opposed components under special conditions.

All these lactones by reduction with sodium amalgam can be converted first into their aldehydes (sugars), and by further reduction into the corresponding alcohols. Upon dissolving $BaCO_3$ in a solution of the lactones, the Ba salt of the corresponding acid is produced, and from this salt the acid can be liberated. In this manner we obtain by reduction from mannonic acid lactone, the corresponding aldehyde (mannose), and the alcohol. These and the acid all polarise to the right. From arabinose-carboxylic acid lactone we obtain a similar series, all polarising to the left, from a mixture of the two lactones a series optically inactive. Hence there can be no doubt that arabinose-carboxylic acid lactone is left mannonic acid lactone, and for the remainder of the paper I shall designate it as such, and also the mixed lactone as inactive mannonic acid lactone.

It has been found that phenyl-hydrazine forms an important series of compounds with several members of the sugar group, and as one of these combinations has proved the key to the solution of the problem of the synthetical formation of the sugar group, it will be advisable to devote a few minutes to a description of these compounds.

Phenyl-hydrazine is a body having the composition Ph[*] $- NH - NH_2$; it possesses basic properties, and readily unites with HCl and acetic acids. It forms with aldehydes and ketones a class of stable bodies [thus with aldehyde the following reaction takes place:—$CH_3COH + Ph - NH - NH_2 = PH - NH - NCH(CH_3) + H_2O$.] When added to an aldehyde member of the sugar group in the proportion of one molecule to one molecule, an exactly similar reaction takes place. Thus, if one molecule of the aldehyde mannose be added to one of phenyl-hydrazine, the compound $C_{12}H\ N_2O_5$ is formed. To this series of bodies Fischer gives the name phenyl-hydrazones or more shortly hydrazones. They are for the most part colourless bodies, mostly soluble in water, and are occasionally of diagnostic value. Under the influence of strong HCl they split up into phenyl-hydrazine and the original aldehyde (sugar).

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[*] $Ph = C_6H_5$, the symbol for the radical of benzene.

When, however, three molecules of phenyl-hydrazine are added to one molecule of an aldehyde or ketone sugar, the following reaction takes place:—$3C_6H\ N_2H + C_6H\ O_6 = C_1\ H_{22}N\ O_4 + C_6H \cdot NH_2 + NH_3 + 2(H_2O)$. In this reaction phenyl-hydrazine plays the part of an oxidiser, two atoms of H being removed; these are not evolved as gas, but react upon the third phenyl-hydrazine molecule, forming aniline and NH_3. To this series the name phenyl-glucosazone or shortly glucosazone has been given. They are difficultly soluble bodies, and are extremely useful in the isolation and recognition of the various members of the sugar group, their melting points, solubility, and crystalline forms, differing. They rotate the polarised ray in the reverse direction to that which the aldehyde of the same series does, and may in this way be used for the purposes of discrimination.

Thus if 0·1 gram. of R. mannose-glucosazone be dissolved in 1 c.c. of strong HC, immediately added to 5 c.c. water and tested in a 10 decimetre tube, it will turn the ray 1·20 to the left.*

A phenyl-glucosazone treated with strong HCl is resolved into phenyl-hydrazine and a body having the composition $C_6H_{10}O_6$, that is, containing two atoms less H than the original sugar. For this class the name glucosone, or, more shortly, osone, has been proposed. They polarise weakly in an opposite direction to the aldehyde sugar of the series to which they belong.

Upon acting on an osone with Zn dust and acetic acid, two atoms of H are attached, and the lævulose belonging to the series is formed. The lævuloses have the formula $CH_2OH \cdot 3(CHOH) \cdot CO \cdot CH_2OH$, and are therefore ketonic bodies. They polarise strongly in the opposite direction to the aldehyde of the same series.

Phenyl-hydrazine also gives a series of very insoluble compounds with the acids of the group; these are distinguished as hydrazides.

Seeing that in one series we have members some of which polarise to the right, some to the left, it is obviously impossible to use the old terms dextro and lævo any longer, as confusion must unavoidably arise. To obviate this, Fischer proposes to name all the series according to the rotation of the aldehyde (sugar) member of the series, and for this purpose use the letters R. L. I. to the compounds in it, just in the same manner as we use O. M. P. in the benzene ring compounds. It is extremely awkward to speak of R. lævulose, and the name "fructose" is proposed instead, which is simply fruit sugar latinised.

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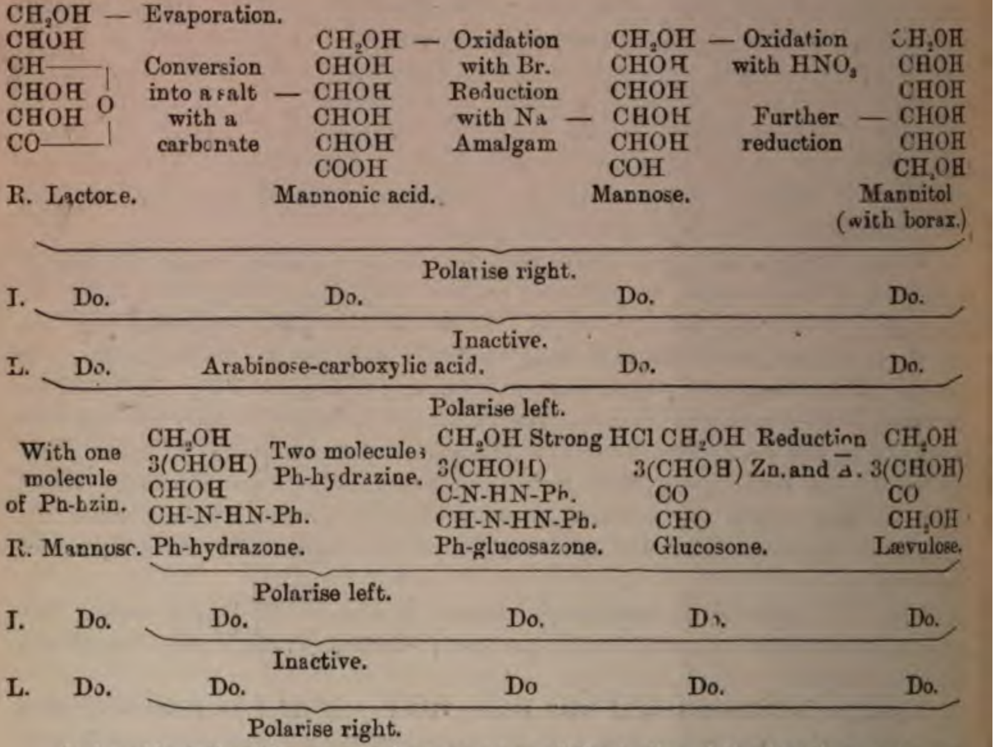
* The observation must be taken quickly, as HCl, even in the cold, decomposes the compound in three or four hours completely.

The accompanying table gives a summary of the group and its various compounds and derivatives :—



R. Lactone.	CH_2OH $CHOH$ CH— $CHOH$ O $CHOH$ CO—	— Evaporation. Conversion into a salt with a carbonate —	CH_2OH $CHOH$ $CHOH$ $CHOH$ $CHOH$ $COOH$ Mannonic acid.	— Oxidation with Br. Reduction with Na Amalgam —	CH_2OH $CHOH$ $CHOH$ $CHOH$ $CHOH$ COH Mannose.	— Oxidation with HNO_3 Further reduction —	CH_2OH $CHOH$ $CHOH$ $CHOH$ $CHOH$ CH_2OH Mannitol (with borax.)
			Polarise right.				
I.	Do.		Do.		Do.		Do.
			Inactive.				
L.	Do.		Arabinose-carboxylic acid.		Do.		Do.
			Polarise left.				
R. Mannose. With one molecule of Ph-hzin.	CH_2OH $3(CHOH)$ $CHOH$ CH-N-HN-Ph. Ph-hydrazone.	Two molecules Ph-hydrazine.	CH_2OH $3(CHOH)$ C-N-HN-Ph. CH-N-HN-Ph. Ph-glucosazone.	Strong HCl	CH_2OH $3(CHOH)$ CO CHO Glucosone.	Reduction Zn. and $\overline{A}$.	CH_2OH $3(CHOH)$ CO CH_2OH Lævulose.
	Polarise left.						
I. Do.	Do.		Do.		Do.		Do.
	Inactive.						
L. Do.	Do.		Do		Do.		Do.
	Polarise right.						

It is evident that if we could form any one of the bodies of the inactive series synthetically from the elements, we could transform it into all the members of that series; and if it were possible to split up any one of these inactive bodies so formed into its optically opposed components, the synthesis of the whole of the members of the group could be accomplished. This has been found possible.

So long since as 1861, Butlerow had observed that formaldehyde in aqueous solution by contact with various bases yielded, by condensation, a sugar-like body, to which he gave the name methylenitan. Loew pursued this investigation further, and found that the various bases differed very much in their power of causing the condensation of formaldehyde. Lead oxide was found to have a remarkably powerful action at a moderately high temperature, even when present in very small quantity. The method which he found to give the best results is as follows :—To four litres of water, containing in solution 40 grams. of formaldehyde, are added half a gram. of magnesium oxide, two or three grams. of magnesium sulphate, and 350 to 360 grams. of granulated lead. The whole is to be kept at a temperature of 60° C. for twelve hours. On evaporation of the fluid, a sweet neutral syrup is left behind, which reduces Fehling, and is capable of undergoing alcoholic fermentation. To this substance Loew gave the name formose.

Fischer obtained, by acting upon acrolein bromide with barium hydrate,* a sugar-body, which he named α. acrose. More recently he was able to form the same body n glycerose obtained in the following manner :—500 grams. of plumbic hydroxide are red intimately into 1,000 grams. of 85 per cent. glycerol; to this is added, with con- t stirring, two and a half litres of ice-cold alcohol. The lead glyceroxide thus formed pread in thin layers, placed under a bell glass in proximity with a shallow vessel taining a quantity of Br equal to half the weight of the lead salt. After the Br is orbed, the product is extracted with alcohol and concentrated in a partial vacuum, n the glycerose remains as a syrup. The syrup is diluted with water and KHO ed until the solution contains about one per cent. of the base, on standing for some s, polymerisation takes place, and amongst other compounds α. acrose is formed.

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* $2C_3H_4Br_2O+2Ba(OH)_2=C_6H_{12}O_6+2BaBr_2$.

He also repeated the above-mentioned Loew's process, and found that Loew's for- e was a mixture of various aldehyde and ketone sugars. Upon treating formose h excess of Ph-hydrazine, a mixture of Ph-glucosazones were formed, all of which e, *with one exception, soluble in alcohol or ether.* The insoluble one, on being treated h strong HCl, yielded the osone, which with Zn and $\bar{A}$ gave α. acrose. This ly, formed by either of the three processes, afforded, upon reduction with Na algam, a body to which he gave the name α. acritol, and which only differed m mannitol* in its optical inactivity. It struck him that this might be ctive mannitol and α. acrose inactive mannose. The first conjecture was doubtedly true, but the second a little removed from the truth, since α. acrose ned out to be a lævulose. The purified α. acrose must naturally, from the de of its formation, viz., the reduction of the osone, be a lævulose; but the original ar might be either an aldehyde or a ketone. Its formation from glycerose led to the w being entertained that it was an aldehyde. Placing two molecules of glycerose to- her, $CH_2OH{\cdot}CHOH{\cdot}COH + CH\ OH{\cdot}CHOH{\cdot}COH$, it would seem most probable that of the H atoms in the second CH_2OH would simply step back and unite with the H group of the first molecule, yielding the aldehyde sugar, $CH_2OH{\cdot}4(CHOH){\cdot}COH$. was found that on treating the original solution in which the sugar had been syn sised with Ph-hydrazine in the cold, no crystalline hydrazone was formed, as is the with mannose, and that only on heating is a glucosazone yielded. This reaction is racteristic for lævulose.

The formation of a sugar allied to grape sugar, by the condensation of formalde- le, is of great interest, since it has been assumed by several botanico-physiologists t this is the first step in the process of assimilation. By the agency of the chloro- ll cells, the H_2CO_3 lost two atoms of O, forming formaldehyde HCOH; three lecules of this polymerise to form a molecule of grape sugar $C\ H_1\ O_6$, which by some ult process loses another molecule of H_2O and becomes starch. The synthesis recently cribed gives considerable confirmation to this view.

If then α. acrose be identical with I. lævulose, either it or one of its derivatives ht to be able to be split up into their optically active components.† Fermentation h beer yeast was found to ferment away the R. portion and leave the L., but here, obviously, the R. mannose is lost, so far as further experimentation with it is concerned. In order to effect the splitting up, the acid was subjected to investigation. The mannonic acid salts of various organic bases were tried, such as cinchonine and cinchonidine, etc.; but all these were found to be too soluble. Strychnine was found to form a mannonate which, upon evaporation of its aqueous solution, deposited itself as a mass of fine crystalline needles. If these be dissolved in absolute alcohol, a clear solution forms for a moment, but almost immediately, at a boiling heat, the difficultly soluble L. mannonate of strychnine falls. This precipitate, upon purification and removal of the strychnine, yields pure L. mannonic acid, and from this all the L. members of the series can be formed.

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* In polarising mannitol and its isomerides, borax was invariably added, which considerably htens their optical activity.

† Lævulose, on reduction with Na amalgam, yields the alcohol of the series, mannitol. From this other members of the series can be obtained in the manner before described.

The separation is, however, by no means complete, the alcoholic mother liquor containing besides all the R. mannonate of strychnine about a third of the L. salt. In order to obtain the R. acid pure, the strychnine is removed from the mixed salts left by the last experiment, and the solution of the mixed acids treated with morphine. From this liquid there separates after long standing crystals in large quantity of R mannonate of morphine. These on purification and removal of the morphine yield R. mannonic acid, agreeing in every particular with the acid obtained from ordinary mannitol. From this acid can obviously be produced all the members of the R. series.

Here then the synthesis of the whole group from formaldehyde is complete, and as this body can be made from methylic alcohol, and the alcohol in its turn be built up from the elements, the synthesis of the whole mannitol group from the elements follows as a natural consequence. The series of reactions involved in this process are grouped in the accompanying table. The way is long and tedious at present, and the percentages of the compound formed in some of the processes very small, but no doubt better and simpler modifications will be discovered hereafter.

From Acrolein Bromide, Glycerose, or Formaldehyde arises,

a. Acrose.

Heated with Ph-hydrazine and Acetic Acid yields

I. Phenyl-glucosazone.

Treatment with strong HCl gives

I. Glucosone.

Reduction with Zn dust and Acetic

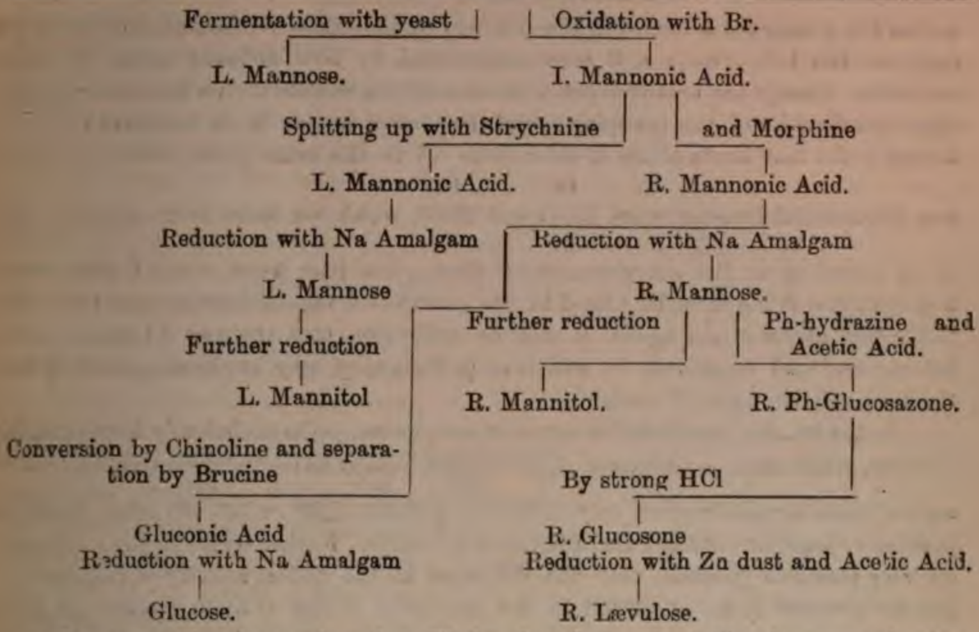
I. Lævulose.

Fermentation with yeast | Reduction with Na Amalgam

L. Lævulose | I. Mannitol

Oxidation with HNO_3

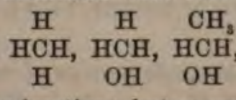
I. Mannose.



I may mention in passing that Fischer found that by boiling ivory-nuts havings with dilute H_2SO_4, they yield about 30 per cent. of R. mannose, and as this ferments readily, he suggests these, which are, at the present time, a waste article, as a source of alcohol.

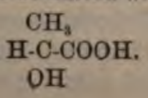
So far nothing has been said about glucose. This body is much akin to mannose. It possesses the same structural formula, forms with phenyl-hydrazine the same glucosazone, which yields the same osone and lævulose. Upon reduction, both aldehydes yield the same alcohol mannitol. Their polarising activities are, however, different. Dextrose turns much further to the right than mannose. The hydrazones are also different; that of the former being soluble, that of the latter being comparatively insoluble. By oxidation with Br both yield acids and corresponding salts. Gluconic acid forms with calcium a salt which separates from a concentrated solution in fine needles, which gradually grow in the fluid into cauliflower-shaped aggregates. This form is characteristic for gluconate of calcium.

It is evident the difference between glucose and mannose must be one of physical isomerism; and for an explanation of this we must turn to the Le Bel and Van't Hoff theory. We know that if a C atom becomes associated with four different groups, it becomes optically active. Thus the molecules



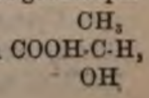
$H\underset{H}{\overset{H}{C}}H$, $H\underset{OH}{\overset{H}{C}}H$, $H\underset{OH}{\overset{CH_3}{C}}H$,

that is, methane, methylic alcohol, ethylic alcohol, are all optically inactive; but so soon as we introduce a fourth group, and cause the C atom to be united with four different groups, we get an optically active molecule, as in lactic acid

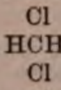
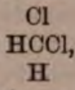


$H\text{-}\underset{OH}{\overset{CH_3}{C}}\text{-}COOH$.

This can be also written



$COOH\text{-}\underset{OH}{\overset{CH_3}{C}}\text{-}H$,

and as the groups are arranged in the one way or the other, so the acid polarises to the right or the left. Such a C atom surrounded by four different groups is called asymetric. Though the arrangement of lactic acid can be shown on a level surface, some other peculiarities of the grouping round the C atom cannot be so exhibited; for instance, if the four bonds of the C atom were all in the same plane, there ought to be two different dichloro-methanes, $\underset{Cl}{\overset{Cl}{HCH}}$ and $\underset{H}{\overset{Cl}{HCCl}}$, which we know there are not. But if, as according to the above-mentioned theory, the four bonds of the C atom are so arranged that if the atom be placed in the centre of a regular tetrahedron, they point to the four angles of the figure, it will be easily seen that the two Cl atoms in the before-mentioned compound, in whatever position they may be placed, invariably hold the same relation to the C nucleus.

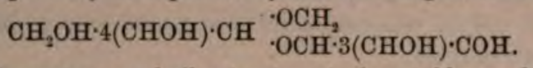
In the hexahydric aldehydes mannose and glucose, we have obviously four asymetric C atoms, which may be designated $CHO{\cdot}\underset{As\,1,}{CHOH}{\cdot}\underset{As\,2,}{CHOH}{\cdot}\underset{As\,3,}{CHOH}{\cdot}\underset{As\,4,}{CHOH}{\cdot}CH_2OH$, and as each of these asymetric atoms may in its turn polarise right or left—in other words, be positive or negative—sixteen modifications are possible. Fischer comes to the conclusion, on very plausible grounds, that the difference in the optical activity of mannose and glucose is caused by an alteration in the properties of the C atom marked As 1, the other three asymetric atoms preserving the same respective relations in the two compounds; so glucose may be looked upon as the right and mannose as the left modification of the same system.

For the purpose of obtaining the gluconic series from the mannonic, the acid seemed the most suitable member to work upon. Mannonic acid may be related to gluconic either as racemic to one of the optically active tartaric acids, or as racemic to meso-tartaric acid. In the first case it should be possible to split up mannonic acid into gluconic and a second isomeride. In the second case there would be more prospect of effecting the transformation by a high temperature. After numerous failures, the conversion was effected by heating mannonic acid with chinoline to 140° C., a mixture of the two acids, mannonic and gluconic, arising, just as a mixture of racemic and meso, tartaric arises upon heating cinchonine racemate to 170° C. After removal of the chinoline, which was liberated by $Ba(HO)_2$, and distilled off by steam, the two acids were separated by conversion into their brucine salts, the mannonic one being insoluble and the gluconic one soluble. After removal of brucine from the gluconate, the acid is evaporated until its lactone forms. This, by reduction with Na amalgam, yields glucose, which has the same specific rotatory power and other characteristics of the natural product. Upon similar treatment the I. and L. mannonic acids should yield an I. and an L. glucose.

Fischer has also made a series of very interesting observations on the saccharons or sugars of the $C_{12}H_{22}O_1$ type, which throw considerable light on the constitution of two of them, amylon and lacton.

Upon treating milk-sugar with Ph-hydrazine and acetic acid the osazone comes down, this by treatment with HCl yields the osone. If this osone be boiled with dilute HCl, inversion takes place, and a mixture of galactose and glucosone in equal molecules arises.

nixture be again treated with Ph-hydrazine and acetic acid Ph-glucosazone comes st, almost in the cold, since an osone combines more readily with this base than an e. Upon warming for some time the Ph-lactosazone comes down. This shows molecule of milk-sugar is built up of a molecule of glucose and one of galactose e COH portion is only free in one group, and this the glucose one, since only group modified by Ph-hydrazine in the milk-sugar molecule before inversion. H group of the galactose portion is evidently altered in the anhydride formation, change may probably be expressed by the following formula:



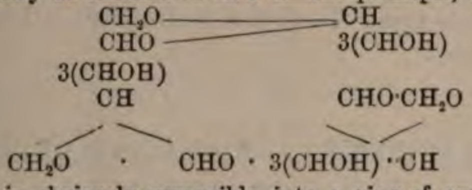
$$\mathrm{CH_2OH\cdot 4(CHOH)\cdot CH}\begin{matrix}\mathrm{\cdot OCH_2}\\ \mathrm{\cdot OCH\cdot 3(CHOH)\cdot COH.}\end{matrix}$$

ylon or malt-sugar upon similar treatment gives evidence of a similar anhydride on between two molecules of glucose.

on treatment with Br lacton and amylon yield two acids of the composition $_2$, called respectively lactobionic and maltobionic. Upon inversion lactobionic ds one molecule of galactose and one of gluconic acid; maltobionic one of glucose of gluconic acid. This is also confirmatory of the structure given above.

e constitution of cane-sugar is still a mystery, if the attempt be made to combine, le of glucose and one of lævulose in a similar manner to lacton or amylon, either yde or a ketone group remains active, and this would necessarily reduce Fehling, ne-sugar does not do. Probably the old view of its being an ether may prove to ct.

oking at the constitution of amylon and lacton, the idea has struck me that and starch may be constituted on a similar principle, thus,



is formula is obviously expansible into a ring of any dimensions, dextrin con- of a smaller number of groups, starch of a much larger. Supposing such a ring groups to be attacked by diastase, and broken up at one point, the ring then maltodextrin, *i e.*, a chain of dextrin groups with an amylon group at each end. in may then be attacked at another point and two smaller chains would arise higher proportion of amylon to dextrin, until at last a number of groups unite form of dextrin on which diastase acts with extreme difficulty.

Killiani's cyan-hydrin process we have a method of proceeding from an aldehyde series to the acid of the one higher up, and in this way can be built up from the ric mannose a heptahydric acid; this can be converted into its lactone, and by n into the hepta-hydric aldehyde. From this, by a repetition of the process, can ed an octohydric aldehyde, and so on. Fischer has in this manner obtained from the heptahydric sugar persite. He proposes to name the aldehydes of the milar in constitution to mannose—mannohexose, mannoheptose, etc., those akin se, glucohexose, glucooctose, etc, the alcohols being expressed by changing the -itol, as mannohexitol, and the acids by changing the -ose into -onic.

On consideration of the results of these experiments, we cannot fail to be struck with some of the curious features of plant life; for instance, all the carbohydrates hitherto examined are members of the R. series; for the members of the L. we have to turn to the gums, and these are looked upon by physiologists to some extent as excretions. They also do not consist of left-handed members only, since O'Sullivan has isolated from a number of gums an arabinose which rotates powerfully to the right. Fischer is inclined to take the view that in plant life, as in artificial synthesis, the inactive series is first formed; this is then split up, the members of the R. series being used up in the formation of starch and the other carbohydrates, whilst the members of the L. serve for some other unknown purpose.

The inability of L. series to undergo alcoholic fermentation is also very curious; bacteria seem to be able to attack R. or L. equally well.

The discovery of some means of producing the lævulose of the L. series (that is, the dextro-rotatory one) on a commercial scale, would be of great advantage to brewers, as it would enable them to produce a beer capable of retaining indefinitely a certain amount of sweetness, a purpose for which saccharin has been proposed. The addition of a carbohydrate sugar would be obviously much less objectionable than the use of a chemical compound, in no way allied to the sugar group except in taste.

Those desirous of pursuing the subject further are referred to the original papers in the "Berichte" of the Berlin Chemical Society, volumes xviii. to xxiii., where the fullest details will be found. Excellent abstracts have also appeared in the journal of the Chemical Society from the year 1887 up to the present time.

In conclusion, I have to thank Mr. Arthur R. Ling for much valuable assistance in the preparation of this paper.

ON SOME POINTS IN THE ANALYSIS OF MILK.

By H. Droop Richmond

(Second Chemist in the Khedivial Laboratory, Cairo).

Read at Meeting, June, 1890.

The importance of the determination of the density of milk cannot be too strictly insisted on, as it affords a most valuable means of checking the results obtained in an analysis, being, in my opinion at least, as valuable as a duplicate determination of the constituents; but there are one or two precautions to be taken in this determination other than those generally applicable to density determinations; as Vieth has recently pointed out (Analyst, XIV., p. 69), the density of milk is not constant until about twenty-two to twenty-four hours after milking, a gradual augmentation taking place. It is the final density which should be taken as the normal density of the sample, as it is the one that has been assumed in the calculation of milk formulæ. Milk is a substance which, when shaken, holds air-bubbles very tenaciously, and sufficient time must always be given after mixing the sample to allow of the elimination of the bubbles; attention to these two points is necessary.

Some time ago I published (Analyst, XIV., 126) a formula for calculating the fat om the total solids and the density (a printer's error in the text gave $T = 1{\cdot}17\,F -$ $63\,\frac{G}{D}$, the real formula being $T = 1{\cdot}17\,F + {\cdot}263\,\frac{G}{D}$). This formula gave practically e same results as that published previously by Hehner and myself (ANALYST, XIII., 26), hich formed the basis of the "milk scale" (ANALYST, XIII., 65). The accuracy cf these rmulæ for cow's milk has been corroborated by some hundreds of analyses in various boratories in different countries.

I have now extended these formulæ to include the calculation of milk-sugar and oteids, the density, fat, total solids and ash being known.

This formula is

$$P = 2{\cdot}8\,T + 2{\cdot}5\,A - 3{\cdot}33\,F - {\cdot}7\,\frac{G}{D}$$

= proteids; T = total solids; A = ash; F = fat; D = density; water at 60° F. being taken as 1; and G = 1000 D − 1000).

The difference between the total solids and the fat, proteids, and ash gives the milk-gar; in this formula it has been assumed that every thing that is not fat, proteids, or h is milk-sugar, an assumption which is not strictly correct, but which introduces a small ror. Another error is probably introduced by the fact that the ash in milk is not the me as the salts existing in milk, but of this I shall speak later; the errors between the oteids and milk-sugar found and calculated vary between + ·4 and − ·4.

From the formula it is easy to calculate the influence of one gram. of each of the nstituents in 100 c.c. on the density and from that the density in solution.

The figures are

	Influence of 1 gram. in 100 c.c.	Density.
Fat	·76	·929
Sugar..	4·00	1·667
Proteids	2·57	1·346
Ash	7·57	4·12

The figures for the density show a remarkable agreement with those directly termined thus. Prof. Fleischmann has found (*J. f. Landw.*, xxxiii, 251) that the erage density of butter-fat is ·93; for milk-sugar many observers agree in giving 1·65; r casein Hehner (ANALYST, VIII., 253) gives 1·31, a number obtained from casein lmittedly not in a state of absolute purity, while from the figures of Dupré (ANALYST, III., 248) I calculate that the density of casein and albumin is 1·34; for the ash I lculate from Dupré's figures (*loc. cit.*) a density of 3·0.

The figures are then thus:—

	Density in solution.	
	Found.	Calculated.
Fat	·93	·929
Milk-sugar	1·65	1·667
Proteids	1·31 (Hehner) 1·34 (Dupré)	1·346
Ash	3·0	4·12

The agreement between the two series of figures is sufficiently exact except in the se of the ash. To this I do not attach much importance for two reasons. It is the gure on which I place least reliance, as a small variation in the factor produces a large variation in the density calculated therefrom, being more than ten times as large as in the other constituents. Then, as Söldner has pointed out (*Landw. Versuch. Stat.* 35, 351), the ash of milk does not accurately represent the salts already existing therein, the citrates and other organic salts being decomposed into carbonates, which are neutralised by phosphoric acid produced by the combustion of the phosphorus of the casein. Neglecting the case of the ash, then, I think there is sufficient proof in the above figures that the density of milk is a simple function of that of its constituents.

The agreement between the two sets of figures also tends to show the accuracy of the methods of analysis. This is especially important in the case of the fat, as doubt has been cast on the method employed (the Adams method as modified by myself); but when we have a formula calculated actually from the results of analysis with almost identical factors to those determined directly, I maintain that it is very strong evidence that the methods employed in the analysis have been correct ones; and the formula is not one for a particular process, but becomes an absolute one.

(*Conclusion of the Society's Proceedings*)

ON THE QUANTITY OF VOLATILE ACIDS PRESENT IN BUTTER FAT.

By Dr. P. Vieth.*

At the meeting held in June, 1889, I laid before our Society the results of a rather extensive series of determinations of the volatile acids obtained from butter fats, treated according to Reichert-Wollny's method (The Analyst, 1889, vol. xiv., p. 147). The present paper is a continuation of that read last year. Some of the results published at that time being rather remarkable, I thought it desirable to extend the investigation and thereby collect more evidence. Last year's paper recorded results up to the 11th of June, and I will now, without further preliminaries, proceed to put before you the results obtained since then until the end of June of the present year.

A sample of pure butter-fat received from Kiel in July, 1888, which on former occasions had required from 31·6 to 32·1 c.c. deci-normal alkali, was repeatedly re-examined, with the following results:

No. 1.	Butter-fat from Kiel	(*a*) 19-9-89	31·8
		(*b*) 12-12-89	32·2
		(*c*) 15-4-90	32·0

This butter-fat has been kept in a well-stoppered bottle protected against the action of light, and does not exhibit signs of decomposition.

I had occasion to examine a large number of samples of butter, as it appears in the market; they are here arranged according to the sources from which they were derived.

Butter Manufactured in London.

No.	Date.		No.	Date.		No.	Date.	
2.	30-7-89	27·1	12.	24-9-89	25·8	21.	8-4-90	29·6
3.	6-8-89	26·0	13.	1-10-89	26·1	22.	15-4-90	28·5
4.	6-8-89	25·9	14.	8-10-89	26·1	23.	22-4-90	27·7
5.	13-8-89	26·2	15.	15-10-89	26·7	24.	29-4-90	28·3
6.	20-8-89	26·5	16.	5-11-89	26·8	25.	6-5-90	29·0
7.	20-8-89	26·7	17.	12-11-89	26·8	26.	13-5-90	28·8
8.	27-8-89	26·8	18.	25-3-90	30·0	27.	20-5-90	28·4
9.	3-9-89	26·3	19.	25-3-90	29·1	28.	3-6-90	28·6
10.	10-9-89	26·0	20.	31-3-90	29·1	29.	24-6-90	27·2
11.	17-9-89	26·4						

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* This paper was intended for the country-meeting, which was abandoned.—P. V

All these results fall within the limits which we are accustomed to look at as normal, viz., from 25 to 30; they are lower in the second half of last, and higher in the first half of the present year.

OTHER ENGLISH BUTTER.

No.	Date.		No.	Date.	
30.	15.4-90	27·3	31.	30-5-90	25·0

Sample No. 31 was butter from a small private dairy, and very rancid when received.

FRENCH BUTTER.

No.	Date.		No.	Date.		No.	Date.	
32.	18-6-89	29·9	45.	10-12-89	27·7	58.	31-3 90	28·7
33.	25-6-89	30·4	46.	17-12-89	26·9	59.	8-4-90	29·0
34.	2-7-89	30·5	47.	31-12-89	28·0	60.	22-4-90	28·8
35.	9-7-89	29·8	48.	7-1-90	28·0	61.	29-4-90	29·7
36.	16-7-89	30·3	49.	14-1-90	27·5	62.	6-5-90	30·0
37.	23-7-89	30·3	50.	21-1-90	27·5	63.	13-5-90	30 5
38.	15-10-89	29·0	51.	28-1-90	28·0	64.	20-5-90	30·8
39.	22-10-89	29·3	52.	4-2-90	27·7	65.	24-5-90	30·5
40.	29-10-99	28·6	53.	11-2-90	28·7	66.	3-6-90	30 2
41.	12-11-89	28·8	54.	18-2-90	28·7	67.	12-6-90	30·6
42.	19-11-89	27·8	55.	25-2-90	29·1	68.	17-6-90	29·8
43.	26-11-89	28 3	56.	4-3-90	29·1	69.	24-6-90	29·4
44.	3-12-19	28·2	57.	11-3-90	29·1			

All these samples contained, as may be seen from the figures, rather large amounts of volatile acids, the results being, with one exception—sample No. 46—all above 27. Much has been said of late of the wholesale adulteration of butter going on in the north of France. Here we have a number of samples which were, no doubt, quite genuine and, I may add, very good in every respect.

DANISH BUTTER.

No.	Date.		No.	Date.		No.	Date.	
70.	22-11-89	29·8	71.	27-11-89	28·8	72.	26-6-90	29·9

The results of the examination of these few samples call for no comment.

KIEL BUTTER.

No.	Date.		No.	Date.		No.	Date.	
73.	18-6-89	28·6	82.	22-10 89	21·7	91.	11-2-90	29·8
74.	25-6-89	27·6	83.	29-10 89	22·6	92.	19-2.90	29·7
75.	2-7-89	25·9	84.	5-11-89	21·1	93.	25-2-90	30·2
76.	9 7-89	26·7	85.	4-12-89	29·2	94.	4-3-90	30·7
77.	16-7 89	27·5	86.	10-12-89	29·3	95.	12-3-90	30·2
78.	23-7-89	24·7	87.	17-12-89	29·8	96.	24-5-90	28 9
79.	30-7.89	27·6	88.	22-1-90	30·2	97.	12-6-90	29·4
80.	6-8-89	24·2	89.	28-1-90	29·5	98.	17-6-90	29·9
81.	15-10 89	22·8	90.	4-2-90	29·7			

The series of samples of Kiel butter comprises some with very low volatile acids. These low results have already been mentioned in a paper which I read before our Society at the February meeting of the present year (THE ANALYST, 1890, vol. xv., p. 44). Great doubts were then expressed as to the genuineness and purity of the samples in question, to which, of course, I cannot swear. But I repeat, most emphatically, my firmest conviction that the samples were entirely free from any sophistication whatever, coming, as they did, from two very well-known and managed dairies, which are entirely beyond suspicion. It is, perhaps, more convincing than my belief to show the peculiar variations which the amounts of volatile acids underwent.

From the one dairy samples were examined at the following dates and with the following results:—

No. 55,	last year's paper	28-5-89	27·3
„ 56	„ „ „	4-6-89	28·7
„ 57	„ „ „	11-6-89	27·4
„ 74	present paper	25-6-89	27·6
„ 75	„ „	2-7-89	25·9
„ 78	„ „	23-7-89	24·7
„ 80	„ „	6-8-89	24·2
„ 81	„ „	15-10-89	22·8
„ 84	„ „	5-11-89	21·1
„ 85	„ „	4-12-89	29·2
„ 86	„ „	10-12-89	29·3

From the other dairy the following results were obtained:—

No. 76	present paper	7-7-89	26·7
„ 77	„ „	16-7-89	27·5
„ 79	„ „	30-7-89	27·6
„ 82	„ „	22-10-89	21·7
„ 83	„ „	29-10-89	22·6
„ 88	„ „	22-1-90	30·2
„ 90	„ „	4-2-90	29·7
„ 91	„ „	11-2-90	29·8
„ 92	„ „	19-2-90	29·7
„ 93	„ „	25-2-90	30·2
„ 94	„ „	4-30-90	30·7
„ 96	„ „	24-5-90	28·9

In the case of both dairies, but more notably in the case of the first one, where there are not so large intervals between the several samples, we notice the volatile acids to drop down to an exceedingly low point during the time from August to November. This is the time when, in Schleswig-Holstein, most of the cows are at the end of the period of lactation. That the butter-fat present in the milk of stale cows contains, as a rule, an amount of volatile acids below the normal limit has been found by several observers, and this fact has certainly something to do with the low results in question. I am, however, inclined to think that there were other natural influences at work, and am glad to know that the matter will be closely investigated this year under more favourable conditions than those under which I had to work.

Investigations executed in various countries have proved beyond doubt that the volatile acids in butter-fat fall below the normal limit much more frequently than was supposed to be the case about two years ago. The fact exists, but as to the reasons we are still very much in the dark. I shall have to add a further number of abnormal cases, but as to their explanation, I am afraid I have nothing to advance.

The samples which I have to mention in what follows are all samples of butter produced in my laboratory by shaking cream in a wide-mouthed, glass-stoppered bottle.

Two samples of cream received from a butter-factory in Sussex gave the following results:—

CREAM FROM GL., SUSSEX.

No.	Date.		No.	Date.	
99.	24-6-89	28·5	100.	10-12-89	28·6

I am now coming to a series of samples comprising abnormal results. The butter- o which all these samples refer was obtained from cream, separated by centrifugal from the milk yielded by cows, which were kept at the Aylesbury Dairy Company's e near Horsham.

I had found last year that the volatile acids in butter-fat produced by the cows kept ıe said estate, were abnormally low during the month of February, March, and part pril; that the results rose up to and above the limit of 25 towards the end of April, that they kept so, though never rising very high, until the early part of June. then the following results have been obtained:—

CREAM FROM HORSHAM.

Date.		No.	Date.		No.	Date.	
18-6-89	26·2	120.	28-10-89	25·4	138.	3-3-90	24·1
24-6-89	26·2	121.	4-11-89	24·8	139.	10-3-90	23·3
1-7-89	25·5	122.	11-11-89	24·8	140.	17-3-90	23·8
9-7-89	25·9	123.	18-11-89	25·1	141.	24-3-90	23·6
16-7-89	25·8	124.	25-11-89	24·0	142.	31-3-90	23·6
23-7-89	25·7	125.	2-12-89	23·9	143.	7-4-90	24·3
29-7-89	26·1	126.	10-12-89	21·7	144.	14-4·90	24·5
5-8-89	25·8	127.	16-12-89	22·6	145.	21-4-90	25·6
12-8-89	25·1	128.	23·12-89	21·3	146.	28-4-90	25·7
19-8-89	24·9	129.	30-12-89	21·7	147.	5-5 90	25·1
27-8-89	25·1	130.	6-1-90	22·1	148.	12-5-90	25·9
3-9-89	23·7	131.	13-1·90	23·1	149.	19-5-90	25·4
10-9-89	23·9	132.	20-1-90	23·3	150.	24-5-90	25·0
16-9-89	23·3	133.	27-1-90	23·2	151.	2-6-90	25·6
24-9-89	23·3	134.	3-2-90	23·1	152.	9-6-90	26·2
30-9-89	23·8	135.	10-2-90	22·6	153.	17-6-90	25·8
7-10-89	24·2	136.	17-2-90	23·9	154.	24-6·90	25·0
14-10-89	25-2	137.	24-2-90	23·6	155.	30-6-90	25·6
21-10-89	25·8						

The figures show that the results kept above 25 until the end of August; that they ped below 25 during September and part of October; that they rose again and kept e, or almost up to 25 till about the middle of December; that they were below om that time until the middle of April; and, lastly, that from the second half pril up to the time at which the present record closes, they were again, slightly gh, but regularly above 25. On comparing the results of last and the present so far as that is possible, one finds that the figures present similar features in fluctuations.

As to the herd of cows yielding in their milk a fat of so frequently abnormal com- ion, it was, in June, 1889, composed of Shorthorn, Jersey and Kerry cows. The eys were eliminated in the latter part of August, 1889, and since that time there about fifty cows milked daily, about three-fourths of that number being Short- s and one-fourth Kerrys. The cows are kept in exceptionally well built, lighted ventilated sheds, which some of our members will, I dare say, remember, from the which our Society paid to the estate in June, 1887. The cows do not calve at one icular time of year; care is, moreover, taken that there are always cows in the various stages of lactation. The food is, for the most part, home-grown, and does not present any unusual features, as, indeed, do none of the conditions under which the cows are kept.

SOME POINTS IN THE DETERMINATION OF SILICA IN SILICATES BY FUSION WITH ALKALINE CARBONATES.

By James P. Gilbert, S.B.

The usual method of determining silica in silicates—that of fusion with the alkaline carbonates, decomposition with hydrochloric acid, and subsequent evaporation to dryness —does not always give satisfactory results. This method is universally described in the text-books as being accurate, with the occasional caution that the filtrate should also be examined for any silica which may not have been rendered insoluble by evaporation to dryness. But one looks in vain for any intimation of what may be the amount of the error, in the determination of silica as usually carried out, if one neglects to examine the filtrate. There is no doubt that many chemists have had trouble with this method, and some have given it up, preferring to volatilize the silica with hydrofluoric and sulphuric acids, and after a careful analysis of the residue, to calculate the silica by difference. A recent writer * makes the statement that he has never been able to obtain more than 97·5 per cent. of the total silica in highly siliceous bodies by the fusion method. The following work was undertaken in the hope of discovering some of the sources of error in the fusion process.

The influence of the temperature at which the silica is dehydrated was first investigated. Most authorities say that evaporation on the water bath to complete dryness, or until no more hydrochloric acid is given off, is sufficient to dehydrate the silica, but a few recommend subsequent heating in an air-chamber to a temperature of 110° C. A higher temperature than 110° C. is said by Fresenius to be inadmissable, since there is liability of the silica recombining with the bases present to form silicates partially decomposable with hydrochloric acid.

To test the influence of temperature on the amount of silica obtained, the following determinations of silica were made in a blast-furnace slag containing about 46 per cent. of lime, 10 per cent. of alumina, and less than one per cent. of magnesia. The silica was dehydrated at various temperatures, but in all other respects the process was carried out exactly as Fresenius directs. What is generally called silica, that is, the mass insoluble in hot hydrochloric acid after evaporation to dryness, is given in Column I. of the following table. This was treated with hydrofluoric acid. and the weight of the residue is given in Column II. In the filtrate from the silica, the alumina was precipitated by ammonia, dried, ignited, and then fused with potassium bisulphate. The residue left on treating the fused mass with water (and correcting for the impurities of the bisulphate used) is given in Column III. The total silica in Column IV. is calculated by subtracting the weight of the residue left after treatment with hydrofluoric acid from the weight of the silica as given in Column I., and adding that found with the alumina in Column III.

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* George Craig: *Chem. News*, Vol LX., No. 1563.

ABLE I.—Analyses of Blast-furnace Slag (containing 46 per cent. Lime) by Fusion with Alkaline Carbonates.

	I. Residue insoluble in hydrochloric acid after evaporation to dryness.	II. Weight of residue obtained from treating Column I. with hydrofluoric acid.	III. Weight of Silica found in Alumina.	IV. Total Silica.
	Per cent.	Grams.	Grams.	Per cent.
1	41·25	·0003	·0006	41·26
2	41·34	·0024	·0008	41·17
3	41·39	·0012	·0009	41·33
4	41·32	·0020	·0003	41·18
5	41·22	·0012	·0009	41·19
6	41·43	·0021	·0007	41·23
7	41·40	·0014	·0009	41·32
8	41·48	·0017	·0008	41·35
9	41·33	·0013	·0005	41·25
10	41·71	·0036	·0007	41·42
11	41·53	·0028	·0007	41·27
12	41·89	·0031	·0004	41·35
13	41·79	·0032	·0002	41·41
14	41·65	·0027	·0005	41·43
15	41·90	·0028	·0001	41·48

Nos. 1 to 3 inclusive were dehydrated on the water bath.

Nos. 4 to 6 inclusive were dehydrated at 125° C.

Nos. 7 to 9 inclusive were dehydrated on iron plate over Bunsen burner.

Nos. 10 to 15 inclusive were dedydrated in air-chamber at 280° C.

All evaporations were made in porcelain.

In this series of results it will be seen that the silica was practically all rendered soluble, even at the temperature of the water bath, and that there was no gain in this spect in using a higher temperature. But the amount of foreign matter in the silica as, in general, perceptibly higher at the higher temperature of dehydration. This is obably due to the alumina being rendered partially insoluble in acid. From the fact at very little silica was found in the alumina, it would seem as if there were no tenncy for the silica to recombine with the lime and alumina even at a temperature of 0° C. Nos. 13, 14, and 15 were digested, after dehydration, with hot acid for one ur, in the expectation of dissolving out all the alumina and other bases, but this eatment did not give smaller residues, after the volatilization of the silica with hydrooric acid, than in Nos. 10, 11, and 12, which were treated only thirty minutes with id.

The general uniformity of the results in this series suggested the possible influence the calcium chloride present in dehydrating the silica on evaporation to dryess. This led to the second point considered, namely, the influence of the salts present on the determination of silica. It is not unreasonable to suppose that some salts, such as calcium chloride, may have a beneficial influence, while others, as, for example, magnesium chloride, which is decomposed when its solution is evaporated, may vitiate the silica determination by forming a silicate which will be subsequently decomposed by hydrochloric acid with solution of the silica. To test the effect of magnesium chloride, determinations of silica were made in a slag containing about 35 per cent. of lime, 15 per cent. of magnesia, and 15 per cent. of alumina. (See Table II.)

Table II.—Analyses of a Blast-furnace Slag (containing 35 per cent. Lime and 15 per cent. Magnesia).

	I. Residue insoluble in hydrochloric acid after evaporation to dryness.	II. Weight of residue obtained from treating Column I. with hydrofluoric acid.	III. Weight of Silica found in Alumina.	IV. Total Silica.
	Per cent.	Grams.	Grams	Per cent.
1	33·65	·0032	·0032	33·61
2	32·90	·0014	·0025	33·18
3	33·46	·0013	·0013	33·29
4	33·71	·0027	·0004	33·40
5	33·42	·0021	·0008	33·24
6	33·75	·0031	·0004	33·36
7	33·49	·0032	·0011	33·24
8	33·85	·0045	·0012	33·27
9	33·90	·0042	·0012	33·30
10	33·65	·0062	·0062	33·62
11	33·83	·0046	·0045	33·80
12	33·95	·0088	·0050	33·60

Nos. 1 to 3 inclusive were dehydrated on the water bath.

Nos. 4 to 6 inclusive were dehydrated at 120° C.

Nos. 7 to 9 inclusive were dehydrated on iron plate over Bunsen burner.

Nos. 10 to 12 inclusive were dehydrated at 280° C.

This series differs from the first mainly in the large amount of impurity in the silica and the large amount of silica in the alumina when the dehydration is effected at a temperature of 280° C. The best results were obtained at a temperature of 120° C. The increase of silica in the alumina as the temperature is increased would seem to be due to the magnesia, for the conditions were, in all other respects, the same as in the analyses of the other slag. The above results indicate that there is no danger of the silica combining with the magnesia at 120° C., and that at this temperature the silica is almost completely dehydrated, the calcium chloride probably assisting in the dehydration.

A series of analyses was next made on orthoclase, practically free from lime and magnesia.

TABLE III.—ANALYSES OF A SAMPLE OF ORTHOCLASE.

	I. Residue insoluble in hydrochloric acid after evaporation to dryness.	II. Weight of residue obtained from treating Column I. with hydrofluoric acid.	III. Weight of Silica found in alumina.	IV. Total Silica.
	Per cent.	Grams.	Grams.	Per cent.
1	64·80	·0018	·0004	64·52
2	64·13	·0018	·0030	64·30
3	64·03	·0026	·0035	64·20
4	64·35	·0020	·0030	64·52
5	64·98	·0050	·0019	64·45
6	64·62	·0035	·0020	64·36
7	64·83	·0068	·0030	64·45
8	64·97	·0083	·0039	64·35

Nos. 1 and 2 were dehydrated on the water bath.

Nos. 3 and 4 were dehydrated at 120° C.

Nos. 5 and 6 were dehydrated on iron plate over Bunsen burner.

Nos. 7 and 8 were dehydrated at 280° C.

This series differs from the previous one in that the higher temperature is not accompanied with an increased amount of silica in the filtrate. This would seem to be a confirmation of the idea that there was a recombination of the silica and the magnesia at the temperature of 280° C. in the magnesia slag; and the fact that the amounts of silica found in the alumina are (with one exception) uniformly higher than those in the lime slag indicates, I think, the beneficial effect of the calcium chloride in the dehydration of the silica.

(To be continued.)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

THE IODINE NUMBER OF URINE AND ITS MEANING. A. JOLLES. (*Wr. Med. Wochenschr. Chem. Zeit.*, No. 15, 1890).—The author calls the iodine figure the quantity of iodine absorbed by 100 parts of urine solids. To carry out the analysis, 10 c.c. of the filtered urine are put into a glass-stoppered bottle, holding about 100 c.c., and mixed with 4 c.c. or more $\frac{n}{10}$ iodine. After standing in the dark for eighteen hours, the liquid is titrated in the usual manner with $\frac{n}{10}$ hypo, adding starch solution at once if the urine was ark-coloured. Care must be taken there is a decent excess of iodine left. The mount of solid matter per litre may be calculated from the formula $T = (s-1)\ 2330$ s representing the specific gravity. If 10 c.c. of urine absorb j grammes of iodine, the iodine figure of the sample $= \frac{10000\,j}{(s-1)2330} = \frac{j}{s-1}\ 4{\cdot}292.$

The following constituents of urine absorb iodine: uric acid, urobilin, and bodies belonging to the phenol type. From the investigation of about 140 healthy urines, the author puts down the iodine figure from 4 to 5·5 The variation chiefly depends on the amount of uric acid.

If the urine is from an unhealthy person, and contains an excess of bilious acids and colouring matters, the iodine figure is much increased, and may come as high as 15. Albumenoids do not absorb any iodine, but in presence of pus the urine will give a very high iodine absorption. It therefore seems the process may prove a valuable addition to the analysis of urine.

L. DE K.

THE KJELDAHL-GUNNING PROCESS FOR ESTIMATING NITROGEN. A. ATTERBERG (*Chem. Zeit.*, No. 31, 1890).—The author strongly recommends this process, a full description of which will be found in the ANALYST, 1889. Even such bodies as quinine and indigotine practically yield all their nitrogen as ammonia. This is no matter of surprise, as in this process the sulphuric acid, instead of getting weaker on boiling, actually gets stronger. No additions of potassium permanganate or mercury, phosphoric anhydride, or scores of other bodies are required; also no potassium sulphide during the distillation.

The author hopes this process will be soon universally adopted in agricultural laboratories.*

L. DE K.

SIMPLE PROCESS FOR THE ESTIMATION OF NITROGEN IN CHILI-SALTPETRE. O. FOERSTER (*Chem. Zeit.*, No. 31, 1890).—When nitrates are repeatedly evaporated with hydrochloric acid, they are gradually converted into chlorides. On this fact the author has founded a process for the assay of nitrate, which, on account of its simplicity and cheapness, will no doubt be adopted. From 2 to 3 grammes of the sample are weighed out (or an aliquot part of the watery solution is pipetted off) and heated to fusion in a weighed porcelain crucible with lid. After cooling and weighing, 25 c.c. of hydrochloric acid (3 volumes of acid, 1 volume of water) are introduced, and the whole evaporated to dryness. This treatment with acid is then repeated twice to expel all nitric acid. The hydrochloric acid must not be too concentrated, otherwise there would be effervescence and spurting. The residue is finally heated for some time to 150° C., then heated to dull redness, and finally weighed. The amount of nitrogen is now calculated as follows: Instead of the molecule NO_3, there comes one atom of chlorine. Therefore the loss in weight is in respect to the nitrogen, as the difference between the molecular weight of NO_3 and the atomic weight of the chlorine to the atomic weight of the nitrogen; therefore, as $61{\cdot}89 - 35{\cdot}32 = 26{\cdot}52 : 14{\cdot}01$, or as $1 : {\cdot}52828$.† The amount of nitrogen is therefore ascertained by multiplying the loss in weight by this factor. It is a pity this process cannot be applied when the sample contains magnesium chloride, as this substance loses hydrochloric acid on drying. The test-analyses are very satisfactory.

L. DE K.

Note by abstractor.—Gunning even succeeded in completely oxidising as much as 100 grammes of meat by his process. Liquids are first evaporated to nearly dryness.—L. de K.

† "Revised Atomic Weights," L. Meyer and K. Seubert.

THE ANALYST.

OCTOBER, 1890

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON THE DETECTION OF HOP SUBSTITUTES IN BEER.

BY A. H. ALLEN AND W. CHATTAWAY.

Read at Meeting, June 1890.

THE method described by one of us in the ANALYST for June, 1887, satisfactorily distinguishes between hops and hop-substitutes in the majority of cases.

It fails sometimes to remove the whole of the natural bitter of the hop, especially in cases where the hops are old. In such cases a better result can be obtained if the excess of lead be removed by boiling with sulphuric acid, instead of by sulphuretted hydrogen, in accordance with the suggestion of Mr. Adams. Concentration in presence of the sulphuric acid appears to be preferable to mere boiling of the dilute acid solution before neutralising with chalk. If the concentrated liquid be agitated with chloroform, most of the bitter principles are extracted (with the exception of chiretin). On evaporating the chloroform solution to dryness, taking up the residue in a few drops of alcohol, adding water, and *tasting* the solution, traces of bitter principles can be detected which wholly escape recognition when the taste of the treated and concentrated beer is observed. Traces of hop-bitter are often detectable when this delicate means of observation is used. According to Dr. James Bell, the extractive matters of the beer interfere with the complete precipitation of the hop-bitter by lead acetate, though that reagent precipitates the bitter principles perfectly from a simple aqueous infusion of hops. Dr. Bell finds that the solvent power of ether on quassia bitter is very limited, while the hop-bitters are readily extracted. Hence on concentrating beer and shaking the acidulated liquid three or four times with ether the whole of the hop-bitter is extracted, while a large portion of the quassia bitter remains in the aqueous liquid and can be extracted with chloroform. If the ethereal solution be evaporated and the residue dissolved and treated with lead acetate, Dr. Bell finds the hop-bitter to be entirely precipitated, while the quassia bitter remains in solution.

We are able confirm Dr. Bell's experience to some extent. That is, we find that if a beer bittered with old hops be clarified by lead acetate, the filtrate extracted by chloroform or ether, and the aqueous solution of the more or less bitter chloroform or ether residue again treated with lead acetate, the remaining hop-bitter is in most cases entirely precipitated; though in the case of one obstinate sample of old hops sent us by Mr. Adams the bitter was not wholly removed even by this supplementary treatment. Our experience is not in accordance with Dr. Bell's so far as the behaviour of quassia with ether is concerned, the bitter being far too easily extracted by ether to render that solvent available as a means of separating quassia and hop-bitters.

With regard to the modified method (Process II.), submitted by the President at the May meeting of the Society, a few experiments made by us have given very satisfactory results. Thus an obstinate sample of old hops, the bitter of which could not be removed by more simple treatment, gave a perfectly satisfactory result when previously treated with baryta-water as recommended by Mr. Adams.

But the advantage gained by a more perfect precipitation of the hop-bitter will be more than counterbalanced if the treatment pursued resulted in the destruction of any of the bitters of the hop-substitutes; and fearing that this might be the case, in our recent experiments we have rather adopted the opposite plan. By so doing, lead precipitation is still relied on as a sorting test, but in the event of a bitter being present which resists the treatment, we prefer to ascertain its nature more fully. This can be done by using the sorting action of chloroform and ether as solvents, and treating the extracts obtained by the evaporation of these with ammoniacal basic lead acetate. This reagent precipitates the bitter principles of old hops and gentian, while leaving those of quassia, calumba, and chiretta in solution. The bitter principles of these substitutes can be recognised to some extent by special tests, which we hope further to amplify and improve; but already we have achieved such a degree of success as to justify us in laying certain of the results before the Society.

Our experiments were first made on decoctions of hops, quassia, chiretta, gentian, and other hop substitutes, and as these proved successful, we proceeded to try the process on mild beer, to which sufficient of the different bittering substances was added to convert it into a fairly palatable bitter beer. Our experiments are not, therefore, open to the objection raised against those of Dr. Bell and Mr. Bannister, described before the Hop Industry Committee. These chemists obtained beer and added quassia and other hop-substitutes in proportions largely in excess of those which would be used in practice, and thus operated under conditions unfairly favourable to their detection.

The following table exhibits the method we have so far found most successful for effecting the separation and detection of bitter principles in beer. By the use of other reagents, especially ferric acetate, a further differentiation can be effected, but we prefer to postpone a description of these experiments till we have verified the reactions at leisure.

OUTLINE PROCESS FOR THE DETECTION OF BITTER PRINCIPLES IN BEER.

A. H. ALLEN AND W. CHATTAWAY.

<table>
<tr><td colspan="6">itre of beer is evaporated to ½ and precipitated boiling with neutral lead acetate; the liquid boiled for fifteen minutes and filtered hot. If any precipitate occur on cooling, the liquid is again filtered.</td></tr>
<tr><td rowspan="4">PRECIPITATE tains <i>hop</i> ter, <i>caramel</i> ter, <i>ophelic</i> <i>id</i> (from retta) phos- tes, albu- ous mat- s, etc., etc.</td><td colspan="5">FILTRATE. The excess of lead is removed by passing sulphuretted hydrogen, and the filtered liquid concentrated to about 150 c.c. and tasted. It is then slightly acidulated with dilute sulphuric acid, and shaken repeatedly with chloroform.</td></tr>
<tr><td colspan="2" rowspan="2">CHLOROFORM SOLUTION on evaporation leaves a bitter extract in the case of <i>gentian</i>, <i>calumba</i>, <i>quassia</i>, and <i>old hops</i> (only slightly or doubtfully bitter in the case of chiretta). The residue is dissolved in a little alcohol, hot water added, and the hot solution treated with ammoniacal basic lead acetate and filtered.</td><td colspan="3">AQUEOUS LIQUID is shaken with ether.</td></tr>
<tr><td colspan="2">ETHEREAL SOLUTION leaves a bitter residue in the case of <i>chiretta</i>, <i>gentian</i>, or <i>calumba</i>. It is dissolved in a little alcohol, hot water added, and the hot solution treated with ammoniacal basic lead acetate and filtered.</td><td rowspan="3">AQUEOUS LIQUID, if still bitter, is rendered alkaline and shaken with ether-chloroform. A bitter extract may be due to <i>Berberine</i> (calumba) or <i>Strychnine</i>.
The aqueous liquid, separated from the ether - chloroform may contain <i>Caramel bitter</i> or <i>Choline</i> (somewhat bitter.)</td></tr>
<tr><td>PRECIPITATE contains <i>old hops</i>, <i>gentian</i>, and traces of <i>caramel</i> products. It is suspended in water, decomposed by sulphuretted rogen, and the solution ated with chloroform.</td><td rowspan="2">FILTRATE is boiled to get rid of ammonia, and treated with a slight excess of sulphuric acid, filtered and tasted. If bitter, it is agitated with chloroform, and the residue examined for <i>calumba</i> and <i>quassia</i>.</td><td rowspan="2">PRECIPITATE is treated with water and decomposed by sulphuretted hydrogen. The filtered liquid is <i>bitter</i> in presence of <i>gentian</i>.</td><td rowspan="2">FILTRATE is treated with a slight excess of dilute sulphuric acid, filtered and tasted. A bitter taste indicates <i>calumba</i> or <i>chiretta</i>, which may be re-extracted with ether and further examined.</td></tr>
<tr><td>HLOROFORM UTION is mined by cial tests <i>gentian</i> and <i>hop</i> bitter.</td><td>AQUEOUS SOLUTION contains traces of <i>caramel bitter</i>.</td></tr>
</table>

In conclusion, we may say that the best way to taste bitter principles is to place e of the solution on the back of the tongue with the aid of a small pipette. When re is sufficient material, some of the solution may be swallowed with advantage. But anterior parts of the tongue are far less sensitive to bitter principles than the parts ards the uvula.

(*Conclusion of the Society's Proceedings.*)

DR. SYKES' PAPER ON THE SUGAR GROUP.

CORRIGENDA AND ADDENDA.

Page 163, line 6, for $C^{12}HN_2O_5$ read $C_{12}H_{18}N_2O_5$.
„ 163, line 12, for $3C_6HN_2H_3 + C_6HO_6$ read $3C_6H_5 \cdot N_2H_3 + C_6H_{12}O_6$.
„ 163, line 13, for $C_1H_{22}NO_4 + C_6H \cdot NH_2$ read $C_{12}H_{22}N_4O_4 + C_6H_5 \cdot NH_2$.
„ 165, line 6 from bottom, for CH_1O_6 read $C_6H_{12}O_6$.
„ 165, line 17 from bottom, for $+CHOH \cdot CHOH \cdot COH$ read $+CH_2OH \cdot CHOH \cdot COH$.
„ 167, middle of page, for H_2SO read H_2SO_4.
„ 168, line 5 from bottom for $C_{12}H_{22}O_1$ read $C_{12}H_{22}O_{11}$.

SINCE the paper was written, Dr. Fischer has succeeded in producing the L and I glucoses. L gluconic acid can be obtained by heating L mannonic acid with chinoline, but the yield is small. It is much more readily obtained by hydrolising the cyanhydrin of arabinose, L gluconic acid and L mannonic acid being formed at the same time. The simultaneous formation of two sterio-isomerides by the addition of HCN to an aldehyde is remarkable, and it is the first time the phenomenon has been observed.

From manno-hexose (obtained by hydrolising ivory nut shavings) (reserve cellulose), the hepta-hydric manno-heptose, gluco-heptose and fructo-heptose have been obtained by the cyanhydrin process. From manno-heptose there have been formed manno-octose and gluco-octose, also from manno-octose manno-nonose. The most of these products exceed in beauty and ready crystallizability the derivatives of the hexose group. Another curious fact has been observed, viz., that only those groups which contain three carbon atoms, or a multiple of three, are capable of undergoing alcoholic fermentation with yeast. Thus the glucerose, hexose and nonose groups only ferment, and this explains the hitherto observed fact that arabinose (pentose) was an unfermentable body.

NOTE ON THE MUNICIPAL LABORATORY IN AMSTERDAM.

By L. DE KONINGH, F.C.S.

SINCE 1879 this laboratory has been managed by a staff of three public analysts (called *Inspecteurs*), one assistant analyst, and ten inspectors (*Keurmeesters*), three of whom take part in the practical analytical work. In England the analyst receives but very few samples which may be considered food proper, but in the above laboratory such foods as meat, fish, poultry, vegetables, preserves, whether in the fresh or prepared state, are occasionally examined. The authorities do not as a rule interfere with the wholesale trade, but take every possible precaution that articles sold retail shall not only be unadulterated, but in a clean state fit for consumption. The inspectors, who report directly to the analysts, and not to sanitary committees, and who are temporary inspectors of police (to enable them to legally seize goods), at once order the destruction of any article which is either damaged by water, fire, or is in a state of decomposition or rancidity. If the shopkeeper insists upon its first being shown to the analysts, of course his wish is gratified.

Eating-houses—even hotels—are occasionally invaded by the inspectors, and we must, I think, admit it would not be a bad thing for the poor if this kind of thing was done over here in some of the less aristocratic quarters.

The power of seizure and confiscation seems to frighten the shopkeepers more than a fine, and is, we believe, a more beneficial measure for the public than which happens in England. Here, for instance, a cheesemonger is prosecuted for selling margarine instead of natural butter. True, when the Court is satisfied he gets a fine heavy enough, but to compensate himself to a certain extent he goes on selling the stuff for a few days, and if he has any left it is mixed up to the extent of say 20 per cent. with pure butter, and so gradually got rid of. He does not entertain the least fear of being caught; as he knows by experience the public analyst will think twice before touching a mixture. This is not due to incompetence, but merely to the fact that if the shopkeeper is at all in a fighting mood he can engage an analyst who may perhaps hold a contrary opinion. No such nonsense in Amsterdam. True, the retailer gets his duplicate sample, but in these eleven years nobody has ever yet come to Court armed with a certificate from an analyst disputing the adulteration, simply because they know the Court is not likely to distrust the certificate of the *legally appointed authorities.*

In cases of grave dispute the case may be referred to some of the professors connected with the University, but their services have not as yet been required. If they ever should we may be sure they would do all in their power to uphold the analyst's certificate.

The appointments are permanent, not as in England yearly ones, and at fixed salaries. We fancy many a public analyst—particularly in the so-called country districts—would not mind being appointed on the same conditions. As matters stand now we know of many public analysts whose income derived from their office amounts to barely a few pounds a year. This is mostly caused by the inspector (who often seems to be the real chief) simply neglecting his duty without any fear of being called to account.

In a laboratory where the officials are possessed of so much power, it becomes doubly important no blunders shall be made; and we fancy that through their system of checking the results not much danger exists in Amsterdam. We will now give our readers a few of the processes and standards in use at that laboratory.

MILK.

The total solids not to be lower that 11·00; fat not less than 3. Consequently Sng 8·0. This standard may seem too low to British analysts, but analyses made by us of genuine Dutch milk by the SPA method fully confirms this standard. As a check on the analyses the specific gravity is always taken, and every sample is put to cream. To better read off the cream the milk is mixed with a drop or two of methyl-violet, which colours the whey, but does not dissolve in the cream. In regard to this important article of food we must confess we prefer the English plan of prosecuting when the milk falls decidedly below standard, never mind its price. In Amsterdam they practically allow any amount of adulteration with water if the price is in agreement with the dilution.*

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*The water used may have been very impure.

BUTTERMILK.

Acidity not to exceed ·3 per cent. The inspectors carry with them a bottle partially filled with a standard ammonia, and coloured with litmus. On adding a definite quantity of the sample the liquid should not turn red. If so there is excess of acid, and without further trouble the supply is at once rendered unsaleable by the addition of some strong colouring matter. Occasionally the samples are taken to the laboratory and tested for excess of salt, showing the use of brackish water.

BUTTER, CHEESE.

Reichert's process. Standard for calculation 12 5 c.c. Every month a butter is prepared of the cream derived from the mixed milk of about 500 cows. Now, if they find this to take say, only, 11 c.c. no butter is touched that month unless it comes decidedly under 11. Water, 16 per cent. of which is allowed, is estimated in Hoorn's apparatus. A weighed portion of the butter is dissolved in benzol, when the water neatly collects in a narrow graduated tube. Salt and caseine, of which there should be no more than 4 per cent. are estimated as usual. Cheese is extracted with ether, and the fat submitted to Reichert's process.

MEAT, SAUSAGES.

The question often arises whether meat, even if apparently still fresh, is likely to soon turn. When meat is distilled with water, the distillate of good meat should remain clear on addition of mercuric chloride. If now a decided precipitate is obtained the sample is at once condemned, and the whole supply destroyed. Hams are constantly examined for trichini, but very few bad cases occur.

BREAD AND FLOUR.

Tested chiefly for alum. The admixture has practically ceased, only two cases having occurred last year.

SWEETS, PRESERVES, AERATED BEVERAGES.

These are all tested for metals, a very small quantity sufficing for their seizure and destruction. One litre of soda-water should leave on evaporation a residue in which neither copper nor lead can be detected. Although some authorities hold that all these articles are much improved by a good, sound dose of copper or lead, no harm has ever come yet from the enforced supply of metal-free aerated water.

SPIRITS, WINES, BEERS.

Spirits are tested for strength, also for fusel-oil, ·2 per cent. of which is allowed.* Should the amount be large the spirit is confiscated. We have been gravely informed some English authorities consider the presence of amylic alcohol highly beneficial; but we can assure the public the grave symptoms of alcoholism are decidedly on the decrease in Amsterdam, thanks to the action taken by the public analysts.

Whether harmless or not, neither salicylic acid nor saccharine are allowed in wines or beers.

PEPPER.

This article when containing excess of husk or stalks is condemned. The analyses are chiefly done microscopically.

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*Qualitatively tested for with sulphuric acid and furfurol, quantitavely by Röse's chloroform *process.*

SOME POINTS IN THE DETERMINATION OF SILICA IN SILICATES BY FUSION WITH ALKALINE CARBONATES.

By James P Gilbert, S.B.

(*Continued from page* 179.)

The third point considered was the influence of temperature on the dehydration of the silica when no bases other than the alkalies were present. From the last three series of analyses it would seem that some silica always fails to be rendered insoluble by heat alone, so that, if a definite amount of pure silica were taken and subjected to alternate fusions and dehydrations, we should expect that the amount of silica obtained would diminish at each successive treatment. To ascertain if this were so, two determinations of silica were made in the lime slag, and the silica thus obtained repeatedly treated by fusion with alkaline carbonates in the usual way. The per cent. of silica recovered in each case is given below. The silica obtained from the slag by the first fusion and dehydration is taken as 100 per cent.

	I. Per cent.	II. Per cent.	
1.	42·02	41·81	Silica in slag obtained by heating to 280°C.
2.	97·24	97·79	Percentage of the above obtained in four successive fusions by heating to 120°C.
3.	96·04	96·70	
4.	95·17	95·96	
5.	94·19	95·45	

The large difference between the results of the first and second fusions is probably due to impurities in the silica, which was heated to 280° C.; but the variations in the other cases can only be due to loss of silica by incomplete dehydration. Still, these are better results than Craig obtained, who does not, however, mention the temperature he used in dehydrating the silica.

The fourth point considered was the possibility of obtaining the silica in a purer state, that is to say, to decrease the amount of the residue left after treatment with hydrofluoric acid. This residue may be due to imperfect washing, or to the fact that alumina has been rendered insoluble in hydrochloric acid by prolonged heating to a high temperature. The higher residues obtained in the cases in which the silica was heated to 280° C. point to the latter possibility. But it is not improbable, as will be mentioned later on, that under certain conditions the silica may enclose alkaline salts so that it is impossible to remove them completely by washing.[*]

In an interesting article by Lindo,[†] on the analysis of glass, I met the recommendation that the watery solution of the fusion be diluted to a very large bulk, so that on acidifying with hydrochloric acid there shall be no precipitation of silica. In this way he obtained, on evaporation, what he called "vitreous silica," in distinction from the ordinary "amorphous silica." This vitreous silica, he says, is so easily washed that time is gained in the analysis to compensate for that lost in the evaporation of the solution. Lindo takes it for granted that one cannot get all the silica by the ordinary process of dehydration, and that the last traces of it cannot be got out of the filtrate unless there is sufficient iron oxide or alumina present to effect its complete precipitation. In the analysis of glass, he added a known amount of ferric chloride to the filtrate from the alumina, and by precipitating this iron with ammonia the last traces were obtained.

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[*] In this connection it is interesting to note that all the residues left on treatment with hydrofluoric acid, in the two slags analysed, contained manganese, which may point to an insoluble compound of manganese with the alkaline salts.

[†] *Chemical News*, Vol. LX., No. 1546.

The following analyses of glass were made according to his directions. The powdered glass, about one gram., was fused with five grams. of sodium and potassium carbonates, and the mass treated with about four hundred cubic centimetres of boiling water until it was thoroughly disintegrated. On acidifying with hydrochloric acid, a clear solution was obtained. This was evaporated to dryness on a water bath, heated to 125° C., and, as recommended by Lindo, the silica thus obtained again fused and treated as before. This silica obtained from the second fusion was weighed, and then treated with hydrofluoric acid. The two filtrates were combined and evaporated to dryness, heated to 125° C. for one hour, and the residue, insoluble in hydrochloric acid and water, filtered off; this is given in the third column. The first three analyses are of German "half-white" glass; the last two of Bohemian white glass.

TABLE IV.—DETERMINATIONS OF SILICA IN GLASS. (LINDO'S METHOD.)

	I. Silica obtained after two fusions.	II. Residue from Column I. with hydrofluoric acid.	III. Silica obtained from the filtrates.	IV. Total Silica.
	Per cent.	Grams.	Grams.	Per cent.
1	73·03	·0003	·0058	73·58
2	71·81	·0008	·0125	73·06
3	72·50	·0004	·0084	73·22
4	71·96	·0000	·0051	71·43
5	71·25	·0007	·0060	71·70

In this case it is seen that a considerable portion of the silica fails to be dehydrated by one evaporation and heating to 125° C. The silica obtained in this way is very compact, and the washing, as claimed by Lindo, is very easily done. The purity of the silica thus obtained (shown by the small residue left on treatment with hydrofluoric acid) is doubtless in part due to the facility with which the silica is washed; for it is conceivable that, when the silica is separated from a concentrated solution of alkaline salts, it may enclose particles of the liquid which may not be easily washed out. But this high purity, as compared with the silica obtained from the slags and the feldspar, may be due to the absence of any large amount of alumina in the glass.

To see what would be the character of the silica obtained from the first slag by large dilution of the solution of the fused mass before acidifying, the following determinations were made:—

TABLE V.—AMOUNT OF FOREIGN MATTER IN SILICA OBTAINED FROM LIME SLAG. (LINDO'S METHOD.)

	I. Weight of Silica obtained from Slag.	II. Residue from Column I. with hydrofluoric acid.	
	Grams.	Grams.	
1.	·2755	·0008	Silica obtained by evaporation to dryness on water bath.
2.	·2610	·0010	
3.	·2871	·0008	Silica obtained by evaporation to dryness and heating to 125°C.
4.	·2683	·0012	
5.	·2506	·0036	Silica obtained by evaporation to dryness and heating to 280°C.
6.	·3113	·0043	

These figures would seem to justify the inference that in the presence of considerable alumina it is not possible, even by large dilution, to get as pure silica as is easily obtained when only salts of the alkalies or alkaline earths are present.

The foregoing determinations confirm the statement that the ordinary process of fusion with alkaline carbonates and evaporation to dryness cannot always be relied on to render all the silica insoluble. No experiments were made to determine the effect of *repeated* evaporations to dryness with hydrochloric acid.

When the evaporation is carried out in the presence of free sulphuric acid the results are more satisfactory, but, as Craig says, the amount of alkaline sulphates introduced seriously interferes with the determination of the bases present.

A comparison of results obtained from a sample of quartz by evaporation with hydrochloric and sulphuric acids is given below:—

I. Silica by evaporation to dryness with hydrochloric acid and heating to 120°. Per cent.	II. Silica by evaporation to dryness with sulphuric acid. Per cent.
98·95	99·43
98·60	99·67
98·79	99·51
	99·70

In these determinations I followed Lindo's recommendation of diluting largely before acidifying, and the residue left, on treatment with hydrofluoric acid, was very small. The first two determinations by sulphuric acid were done by decomposing the dilute watery solution of the fusion with hydrochloric acid, evaporating to dryness, and then adding strong sulphuric acid in excess and heating on an iron plate until copious fumes of sulphuric acid were given off for several minutes. The silica was in appearance the same as obtained by Lindo's method with the aid of hydrochloric acid only. But in the last two the watery solution of the fusion was decomposed directly by sulphuric acid in considerable excess, and then evaporated as before. The silica obtained in this way was very bulky and gelatinous.

These results seem to justify the recommendation that has been made, namely, that in the analysis of silicates the silica is best determined by dehydration with sulphuric acid (in cases where it is not inadmissible by the presence of lime, barium, lead, etc.), and that the bases should be determined, after the decomposition of the silicate, by hydrofluoric acid.

ESTIMATION OF FUSEL-OIL IN SPIRITS.

By A. Stutzer and O. Reitmair.*

In our former paper on this subject (Rep. *Anal. Chemie.*, 1886), we gave it as our opinion that of the processes from time to time proposed for the determination of fusel-oil in spirits, only two are reliable, viz., the process of Röse (as modified by us), and the capillarimeter of J. Traube. We then observed that the last process may give too high results if the brandy contains large quantities of ethereal oils. Several colleagues confirmed our experiments and adopted our final process. The minute investigations of E.

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* *Zeitschr. f. angew Chemie*, Sept., 1890.

Sell also showed our process to be preferable to all others. True, the analysis is easier and more quickly performed with Traube's capillarimeter, but when the spirit is very poor in fusel-oil, the results with Röse's method are more exact. As regards the more recent process recommended by Traube, and called the stalagmometric method, Sell thinks it to give results less reliable than the two just mentioned.

W. Fresenius has recently published a research on the estimation of fusel-oil in spirits of wine, cognac, arrac, and other strong spirits. He used by preference Röse's method with a large shaker, but also employed Traube's two apparatus. Fresenius does not say which of the three processes is the most reliable, but believes the stalagmometer to be the more suitable for such analysts who but rarely have to estimate fusel-oil in spirits. Noteworthy is Fresenius' remark that supposed pure commercial spirits showed a different behaviour, and he says: "Apart from experimental errors, this can only be explained by some spirits containing a substance with properties reverse to those of fusel-oil." If this idea should be confirmed, the whole principle of the two methods would be rudely shaken, because it is always as well to do a check with pure spirit; but what is worse still, it would be possible for a sample to contain a large quantity of fusel and still not show any at all, by the physical processes.

We have once more studied the matter and made it our task to estimate the fusel, not only in brandies (branntweinen), but also in the stronger spirits, with greater accuracy than before. For the testing of spirits, the process adopted for brandies is not sufficiently accurate. It must be remembered that the refining value of spirits is much influenced by minute quantities of fusel, and as the spirit has to be diluted up to 30 or 20 per cent., the experimental errors will be about three times as great as with a sample of brandy with say 30 per cent. of alcohol. E. Sell is certainly of opinion that in the Röse-Herzfeld's apparatus ·01 c.c. of increase in the chloroform may be read off with certainty; but, calculated on absolute alcohol, this means ·022 per cent. of fusel-oil. As, however, with strong spirits, the error may be two or three times higher, it might easily happen that one analyst declares a sample pure and another finds as much as ·1 per cent. of fusel. In our recent investigations we made use of Traube's capillarimeter and also of Röse's shaking apparatus. We thought of getting, in the case of brandies, the best results with Traube's process, because it is not interfered with by ethereal oils. For Röse's process we used the new apparatus of W. Fresenius, which we have seen at work in the laboratory at Wiesbaden. A graduation of the small tube in 0·2 of a c.c. has its drawbacks, as the chloroform collects but slowly in the small tube, which is also difficult to clean. As besides, the larger apparatus is 2·5 times more accurate, an increase of 0·5 c.c. means the same as a ditto of ·02 c.c. in the small Windish's apparatus. A more accurate estimation of the fusel-oil was made possible by first removing part of the pure alcohol, and so getting a product richer in fusel. This succeeded best by a fractional distillation, sometimes with addition of calcined carbonate of potash to retain the water. In cases where the fusel did not exceed ·1 per cent. we succeeded in getting a tenfold concentration.

The boiling-point was but little higher than that of absolute alcohol, and remained constant almost to the end of the distillation. The process of distilling will be presently described; the distillates contained a percentage by volume from 95–96.

General Remarks About the Use of the Large Shaking Apparatus.

For the calculation of the amount of fusel, we proposed to take the difference between the increase of the chloroform when shaken with a *pure* alcohol and with the sample, and found that the presence of ·1 per cent. of amylic alcohol caused an increase of ·15 c.c. in the volume of the chloroform. If the temperature exceeded 15° C., we proposed a decrease, or increase, of ·1 c.c. The Government officials, however, were directed not to use the correction for temperature, but to make the experiment at exactly 15° C. We, however, think it best to make a correction at all events if the temperature is very near 15° C.

In using the large shaker, the chloroform, of which we use 50 c.c., should be of a temperature of 15° C.; if possible the 250 c.c. of the alcoholic distillate (of 965·6 sp. gr.) of the same temperature. The refrigerating fluid is most useful when a few tenth under 15° C., and this must not alter during the experiment. In this manner the temperature of the chloroform is sure not to differ much from 15° C.

A. *Correction for Temperature.*—We have made several experiments in this direction. 50 c.c. of chloroform were put into the apparatus, 250 c.c. of the alcoholic fluid were introduced, and finally 2·5 c.c. sulphuric acid of 1·286 sp. gr.* After thorough shaking, we waited until the chloroform had completely separated, and then put in the thermometer, which showed 15·2° C. The volume of the chloroform was now 54·7 c.c. The shaking was several times repeated and the volume of the chloroform again noted at various temperatures.

Temperature	15·5° C.,	volume	chloroform	54·75 c.c.
„	15·8°	„	„	54·80
„	14·1°	„	„	54·58
„	14·4°	„	„	54·61
„	14·7°	„	„	54·66
„	14·9°	„	„	54·68
„	15·0°	„	„	54·69
„	15·3°	„	„	54·72
„	19·3°	„	„	55·56
„	19·4°	„	„	55·56

Disregarding experimental errors, the correction for the greatest difference in temperature (4·4° C.) is ·87 c.c.; consequently ·2 c.c. for every degree between the temperatures of 15° and 15·8° C., the allowance is ·014 c.c. for every ·1° C.; between 14·1° and 15° C. it is ·012 for every ·1° C.; and for the still lower temperature it only amounts to ·01 c.c. for every degree alteration in temperature.

In similar manner, several other experiments were carried out, and finally the correction of ·01 c.c. for every degree difference in temperature proposed, provided the temperature does not sensibly vary between 14·5 and 15·5° C. The shaking and reading off must be at least twice repeated.

B. *The Check with Pure Alcohol.*—Three samples of alcohol from various sources, the first two bought as pure and absolute, and the third as a pure 96 per cent. spirit, were each experimented upon.

The chloroform was bought as pure, and was not subjected to any purification in our laboratory. The watery solution gave a very faint iodoform reaction. The alcohols were at once diluted up to 30 per cent. without previous re-distillation, ·1 per cent. strength being considered as within experimental error. The volume of the chloroform in three shakings did not differ more than ·03 c.c.

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* Addition of sulphuric acid slightly prevents the solution of pure alcohol in the chloroform.

		Volume of chloroform 15° C.
A purest absolute alcohol	...	54·63 54·64
B ,, ,, ,,	...	54·60 54·60
C pure 96 per cent. alcohol	...	54·62 54·65 54·64

The same alcohols were now re-distilled with pure KHO, and the rectified product again shaken.

A	...	...	54·63
B	...	...	54·60

1,000 c.c. of C alcohol were then put into a two-litre retort and slowly distilled from a sandbath, 100 c.c.'s being separately collected. After the distillation was over, about 300 c.c. of water were put into the empty retort, and again distilled until the boiling-point had become constant. The difference between 100° C. and this constant boiling-point was added to the boiling-points of the various fractions, so as to get thorough comparable results unaffected by barometrical pressure variations. The watery distillate was united with the last spiritual fraction. The boiling point of the alcohol kept almost constant (78·6), but only at the very last it got up to 79·5.

The different fractions were now each brought up to 965·6 specific gravity and then shaken out with chloroform, with the following results:

Fraction.	Temperature 15° C.	Average.
1	54·82 c.c.	54·74
2	54·69 ,,	
3	54·74 ,,	
4	54·71 ,,	
5	54·57 ,,	54·51
6	54·53 ,,	
7	54·50 ,,	
8	54·47 ,,	
9	54·50 ,,	
10	54·72 ,,	
Actual average		54·625

The fractions from 5 to 9, therefore, gave a decided lower chloroform figure than the first and last fractions, and it seemed, therefore, that our so-called pure alcohol contained impurities. The alcohols A and B were now also submitted to fractional distillation, and the sixth and tenth fractions tested:

		Sixth fraction.	Tenth fraction.
A	..	54·50	54·69
B	..	54.50	54·70

From these experiments it is plain that we cannot trust to the purity of commercial alcohol, but that the test-alcohol must be purified by fractional distillation and then tested with the chloroform.

C. *The Increase of the Chloroform Volume by Amylic Alcohol.*—Before attempting to further improve on the method, we first wanted to test its present accuracy. Of alcohol A, a large quantity was diluted up to 30 per cent. by volume, and a mixture was also prepared containing 1 per cent. of amylic alcohol by volume, and which also showed a sp. gr. of 965·6 at 15° C. By mixing the two spirits we got mixtures with definite amounts of amylic alcohol. The shaking was performed as described with chloroform and sulphuric acid.

Per cent. of Amylic Alcohol.	Volume.	Chloroform.
·0	54·64	54·63
·02	54·72	54·71
·04	54·78	54·78
·06	54·84	54·85
·08	54·91	54·92
·10	54·99	55·00
·20	55·36	55·35
·50	56·48	56·47
1·00	58·32	58·31

·01 c.c. increase in the chloroform, therefore, corresponds with ·0028 per cent. of amylic alcohol. The used apparatus, therefore, is capable of determining ·02 per cent. of amylic alcohol, because the difference in chloroform volume for ·02 c.c. amylic alcohol = ·074 c.c. But on the other hand, an experimental error of ·01 per cent. must be allowed for. The liquid actually experimented on occupies about three times the volume of the original alcohol. Therefore, under the most favourable conditions, ·06 per cent. may be estimated with fair accuracy in the stronger spirits. And sometimes we may even be in doubt whether such a spirit may not contain ·1 per cent., if the check-spirit has not given a very favourable result.

(*To be continued.*)

THE USE OF ANIMAL CHARCOAL IN THE DETERMINATION OF FAT (ETHER EXTRACT) IN FEEDING STUFFS.

By H. J. Patterson.[*]

The crudeness of the product which goes under the term "ether extract" in fodder analyses is a thing that has impressed itself upon all who have to any extent been engaged in the determination of the various constituents of farm products. How to get rid of a portion, if not all, of this extraneous matter, and have nothing but fat or very closely allied products, has been an interesting question, up to this time unsolved.

To obviate this difficulty I have successfully used animal charcoal, and have obtained a product which in many cases was nearly pure vegetable oil, and in no instance could much foreign matter be detected, getting in every case a product having the general appearance and characteristics of pure fats.

The preparation of the sample and manner of treating the extract has been precisely that adopted by the Association of Official Agricultural Chemists. In filling the percolator tube there was first placed in it the animal charcoal, on top of this a plug of cotton, then the substance to be extracted, and finally another plug of cotton. The cotton serves to make a more uniform stream of ether over the substance, and keeps the latter from crawling up the sides of the percolator.

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[*] "American Chemical Journal."

The plug between the substance and the charcoal will prevent any of the fat coming in contact with the charcoal before it is thoroughly in solution. In a few instances, especially in the case of grains, it was found that the results were a little too low if the fat came in contact with the charcoal before it was thoroughly held in solution by the ether.

In all cases one gram. of the substance was used. With grains and meals one gram. of charcoal was used, and with hay, fodders, and the like, two grams. The amount of charcoal can be varied at the discretion of the analyst, but the above amounts were found satisfactory. The animal charcoal was a pure article, of medium fineness, thoroughly dried, extracted with ether, and again dried and preserved for use in a well-stoppered bottle. The cotton was of good quality and clean, and had been extracted with ether previous to use.

In the following discussion the method of the Association of Official Agricultural Chemists is designated as Method I., and where charcoal has been employed as Method II.

To prove that there is no loss in passing of pure fat through charcoal, a number of fats were absorbed on cotton and extracted by each of the two methods. The results are given in the following table (A.):—

TABLE A.

	METHOD I.			METHOD II.		
Sample.	Fat Added. Gram.	Fat Recovered. Gram.	Loss or Gain. Gram.	Fat Added. Gram.	Fat Recovered. Gram.	Loss or Gain. Gram.
Cotton-seed oil,	·2180	·2180	0000	·1770	·1760	—·0010
,, ,,	·2020	·2025	+·0005	·2015	·2010	—·0005
,, ,,	·2015	·2010	—·0005	·1815	·1810	—·0005
Average,	·2072	·2072	0000	·1867	·1860	—·0007
Butter,	·1775	·1770	—·0005	·1765	·1760	—·0005
,,	·1520	·1510	—·0010	·1880	·1870	—·0010
,,	·0910	·0910	0000	·1030	·1035	+·0005
Average,	·1402	·1397	—·0005	·1558	·1555	—·0003
Mutton tallow,	·1160	·1165	+·0005	·1470	·1480	+·0010
,, ,,	·0960	·0960	0000	·0925	·0930	+·0005
,, ,,	·0970	·0970	0000	·0810	·0815	+·0005
Average,	·1030	·1032	+·0002	·1068	·1075	+·0007
Lard,	·0675	·0670	—·0005	·1415	·1420	+·0005
,,	·1345	·1340	—·0005	·0925	·0930	+·0005
,,	·2330	·2325	—·0005	·0660	·0660	0000
Average,	·1450	·1445	—·0005	·1000	·1003	+·0003
Beef tallow,	·1100	·1100	0000	·1125	·1120	—·0005
,, ,,	·1515	·1520	+·0005	·1545	·1540	—·0005
,, ,,	·0965	·0965	0000	·1335	·1335	0000
Average,	·1193	·1195	+·0002	·1335	·1332	—·0003
Sum of averages,	·7147	·7141	—·0006	·6828	·6825	—·0003

From this table it will be seen that there is no appreciable loss, and that the two methods are about the same when pure fats are being extracted. The different fats employed give about as much range in composition and physical characters as would be expected in the various vegetable fats.

In Table B. is given a comparison of the two methods on a variety of samples, which cover fairly well the different classes of substances met with in agricultural analysis. A comparison of the results given in table B. shows that there is a difference in every case, Method I. giving the higher result, and in some cases the difference is surprising. This shows that the amount of error which has been introduced into our valuations of feeding stuffs, calculation of digestibility and nutritive ratios, is very much more exaggerated and misleading than has been supposed. The results in Table B. are the average of triplicate determinations.

TABLE B.

Description of Sample.	I. Per Cent. Fat.	II. Per Cent. Fat.	Difference. Per Cent.
Clover hay	3·51	2·02	1·49
Corn fodder	6·17	2 69	3·48
Corn meal	4·63	4·53	0·10
Cotton-seed meal	10·50	9·88	0·62
Dung from digestion experiment on cotton-seed meal	5·83	3·83	2·00
Oats hay	4·12	2·57	1·55
Soja bean silage	5 53	4·70	0·83
Sorghum fodder, "Early Orange" ..	5 46	2·70	2·76
Sorghum silage, "Amber"	6·15	3·68	2·47
Tomatoes (dried)	10 07	6·48	3·59
Unknown pea	3·44	1·76	1·68
Wheat bran	3·80	3 33	0·47

Having noticed the acidity of the smell of the extract of some of the samples and of ether which was distilled from the extract of some plants, a few tests of the amount of acid in the extracts, soluble in cold water, were made by titrating it with decinormal sodium hydrate, phenolphthalein being used as an indicator. In nearly every case some acid was found in the extract of Method I., reaching over 1 c.c. in some cases, there being scarcely a trace from the extract of Method II. The only case in which there was an appreciable amount of acid in the extract of Method II. was from the sorghum silage.

From a study of these figures and a close examination of the products obtained by the two methods, it seems that the use of charcoal results in a closer approximation to the truth than any other method in use, though absolute accuracy is not claimed. The following points may be rightly claimed in favour of the use of animal charcoal in the determination of fat (ether extract) in feeding stuffs.

(1) That the product obtained is nearly pure fat or vegetable oil.

(2) That the product obtained gives a more correct idea of the physical nature of the fats from various substances.

(3) That slight quantities of water that may exist in the substance and pass out with the extract will be removed by the charcoal.

(4) That soluble acids of the plant, or acid which may be formed by the continuous distillation of ether, in connection with some constituents of plants, will be partially, if not wholly, removed by the animal charcoal.

(5) That the animal charcoal will partially obviate, if not wholly remove, the difficulty of change in the amount of ether extract (which generally increases) with the aging of the sample.

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REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

Analyses of East Indian Honeys. Van der Marck. *Ned. Tydschr. v. Pharmacie*, etc., September, 1890.

Name.	Spec. gravity at 12° C.	Colour.	c.c. of a 10 per cent. solution by volume required to reduce 10 c.c. Fehling.		Polarisation of 10 per cent. solution in 20 c.m. tube.	Ash per cent.
			Before inversion.	After inversion.		
—	1 3134	Yellow	9	8·5	+ 13′	·26
Mengkepar ..	1·3546	Brown	7·2	7 2	−2°54′	·39
Plarvan ..	1·3426	Dark Brown	7·6	7·6	− 52′	·54
Pœlas ..	1·3099	Yellow	8·0	8·0	− 3′	·12
Tjeret Madce ..	1·3569	Light Brown	6·9	6·9	−1°53′	·43
Tarvon Blcra ..	1·3586	Very Dark Brown	6·7	6·7	−1°36′	·39

Applying the usual equation, the author finds the composition

	Glucose.	Levulose.
of No. 1 ..	28·70 ..	22·90 (in addition 3 per cent. of saccharose.)
„ 2 ..	24·60 ..	39·60
„ 3 ..	31·11 ..	29·99
„ 4 ..	32·90 ..	25·20
„ 5 ..	30·80 ..	36·60
„ 6 ..	33·30 ..	36·62

The acidities of the samples were respectively 1·8, ·1, ·2, ·2, ·2, ·4 c.c. $\frac{N}{10}$ soda. Their ashes required ·6, 2·4, 3·6, ·1, 3·0, 2·6 c.c. $\frac{N}{10}$ oxalic acid.

L. de K.

ESTIMATION OF ZINC IN SILICEOUS ORES CONTAINING LEAD. DR. W. MINOR. (*Chem. Zeit.*, No. 61).—Two grams. of the ore are boiled for fifteen minutes with 60 c.c. of weak soda ley. After pouring off, the insoluble matter is again boiled with soda. The liquid is filtered off and the residue washed with boiling weak soda. The filtrate is acidified with sulphuric acid, which causes any lead to separate as sulphate. Water is now added up to a definite bulk, and after standing for a few hours, half of the volume is filtered off, and after mixing with excess of ammonia titrated with standard sodium sulphide. No notice need be taken of the little alumina which generally forms on adding the ammonia. By this process we only get the zinc in combination with carbonic or silicic acid, but not the sulphide. L. DE K.

ASSAY OF RAW MATERIALS CONTAINING TARTARIC ACID. DR. LAMPERT. (*Chem. Zeit.*, No. 55).—This analysis is not always reliable. The author mentions an instance where the same sample was analysed five times by a leading analyst and gave results varying from 23 to 33 per cent., whilst differences of 2 or 3 per cent. are quite common when the assay is performed by Goldenberg's process. This process in itself is exceedingly accurate, so the bad results sometimes obtained have been ascribed to the presence of organic matter. But although pectin for instance may influence the result a little, it is not to be held responsible for the serious differences just mentioned. The author after a long investigation at last found the enemy to be alumina, which if present in large quantity entirely upsets Goldenberg's process. Experiments on known mixtures showed a deficiency of 5 per cent. of tartaric acid when the mixture contained 25 per cent. of alum, 9·5 per cent. when 45 per cent. of alum was present, and 15·5 per cent. if the alum reached 50 per cent. The author also found the reason why the same chemist got such varying results with the same sample. It is because the loss in tartaric acid not only depends on the amount of alumina but also on the amount of acetic acid used. The author has invented a process which almost entirely removes the alumina from tartaric liquors. Liquids so treated showed 80 per cent. of tartaric acid instead of 49, and no serious differences were noticed on submitting the same sample to different analysts. Unfortunately the author does not give any details of his important process. L. DE K.

ARSENIC TEST.—The behaviour of the hypophosphites towards arsenical solutions (precipitation of metallic arsenic) is brought into prominence again by G. Looff as a test for arsenic; in sensitiveness it ranks between the tests of Gutzeit and Bettendorf. Ten c.c. hydrochloric acid, of any strength or purity, with 0·2 gram. calcium hypophosphite placed in a water-bath for 1-2 hours, will enable the detection of $\frac{1}{100}$ milligram. arsenic (Bettendorf's test will detect $\frac{1}{50}$ milligram.). Five c.c. sulphuric acid, phosphoric acid or glycerin, with 0·2 gram. of a hypophosphite (in the presence of sulphuric acid the sodium salt is best used, in other cases the calcium salt, because of its non-hygroscopic character) and 10 c.c. concentrated hydrochloric acid warmed in a water-bath for 1-2 hours will show the presence of $\frac{1}{80}$ milligram. arsenic. Of calcium phosphate, sodium phosphate and tartar emetic, 0·5 gram. is dissolved in 10 c.c. concentrated HCl and 0·2 grams. calcium hypophosphite added, etc. Bismuth subnitrate must first be ignited to remove the nitric acid, and then proceeded with as above; or dissolve equal weights of the subnitrate and hypophosphite in hydrochloric acid and warm. Sulphide of antimony is dissolved in HCl with the aid of a little potassium chlorate before adding the hypophosphite and warming. To test solution of ferric chloride, a combination of Bettendorf's test and the hypophosphite test allows the detection of the smallest trace of arsenic: 5 c.c. of the solution mixed with 10 c.c. HCl are decolorised by addition of Bettendorf's test solution, then the hypophosphite added and warmed. Most of the commercial solutions of ferric chloride were found to contain arsenic.—*Am. Journ. Pharm.*

A Morphiometric Assay of Opium, for which is claimed purity of the morphine obtained, the alkaloid being almost white and immediately soluble in 100 parts lime water, considerable saving of time, and, constant results, the extreme differences of a number of essays being within 0·3 per cent. (the results of this method are always about 1 per cent. lower than by Dieterich's method) is as follows: 5 grams. of the finely powdered opium are triturated with water and made up to 78 grams.; after frequently agitating during 1-2 hours, 60·8 gram. (representing 4 grams. opium) are filtered off, and in it dissolved 0·2 gram. oxalic acid. After one half-hour, 5·2 grams. of a solution of potassium carbonate (1 : 2) are added, thoroughly mixed (avoiding unnecessary agitation) and 16·5 grams. filtered at once through a dry plaited filter of 12 cm. diameter into a tared flask of 30 c.c. capacity. To the 16·5 gram. filtrate (representing 1 gram. opium) add 5 grams. ether free from alcohol, cork the flask and agitate briskly for ten minutes; the ether is then evaporated by use of a small rubber blast, the morphine collected on a small plain filter and thoroughly washed with water saturated with ether, dried at 40-50° C., returned to the flask, which has been dried in the meantime, and weighed to constant weight.

The addition of the oxalic acid is made to precipitate calcium salts, which are present in all opium varieties excepting Salonica opium, which gives no perceptible precipitate; by the use of a large excess of potassium carbonate the narcotine is completely and immediately precipitated, while no morphine is precipitated in the minute's time necessary to filter off the 16·5 gram. filtrate.

This method has also been applied to the examination of *extract* and *tincture of opium*. 2·5 grams. *extract* are dissolved in water, with the addition of 0·2 gram. oxalic acid, diluted to 70 grams., 5 grams. solution of potassium carbonate added, 15 grams. filtered off (corresponding to 0·5 gram. extract., etc., as above).

Fifty grams., with 0·2 gram. oxalic acid, are evaporated to a thin extract and gradually diluted with water to make 70 grams.; to this add 5 grams. solution of potassium carbonate, and filter off 15 grams. (corresponding to 10 grams. tincture), and proceed as above.—*Am. Journ. Pharm.*

The Detection of Nitrobenzol or Oil of Mirbane in oil of bitter almonds succeeds easily by warming the suspected oil with black oxide of maganese and sulphuric acid. Nitrobenzol does not lose its odour by this treatment, on the contrary, the odour becomes more pronounced, after standing for awhile an odour of oil of cinnamon is developed; oil of bitter almonds at first develops a disagreeable odour, which, after some time, entirely disappears.

To detect nitrobenzol in soaps, solutions, etc., soaps are first dissolved in water ; the solutions are treated with an excess of slaked lime, extracted with ether, the ethereal solution evaporated to dryness on a water-bath, and the residue shaken up with a little water. In a small porcelain capsule are placed two drops liquefied carbolic acid (made by adding 10 parts water to 100 parts of the crystallised acid), three drops distilled water, and a piece of potassium hydrate of the size of a pea. This mixture is heated to the boiling point, care being taken to prevent charring of the mass, and a few drops of the ethereal residue mixture added; on continued boiling, a carmine-red ring is formed around the edge of the liquid, the depth of colour depending upon the quantity of nitrobenzol present; the addition of calcium hypochlorite solution changes the red into a beautiful green colour.—J. MORPURGO, *Pharm. Post.*

CORRESPONDENCE.

[*The Editor is not in any way responsible for opinions expressed by his correspondents.*]

To the Editor of the ANALYST.

SIR,—During the last few months considerable attention has been attracted among public analysts to the largely-increasing substitution by grocers of an imitation article for the well-known Demerara yellow crystals, and as considerable misconception appears to exist in analytical circles as to these, it has occurred to me that a few words on the subject from one who has been closely associated with the manufacture of the real product for some years may not come amiss to your readers.

The term " Demerara crystals " is applied to the yellow crystallised grocery sugar coming from the colony of British Guiana (of which Demerara forms a part). Recently a similar sugar has also been produced by some of the West India Islands, where the Demerara process of manufacture has been adopted.

The peculiarity of this sugar is that, although it may be said to be refined—containing, as it does, from 95 per cent. upwards of sucrose, according to the degree of dryness required by market considerations, the characteristic smell and sweetness of the cane juice has been to a large extent preserved. To secure this end, a process has to be adopted of a special nature, and a product is thus obtained which on account of its possessing the attributes mentioned above, commands a relatively high price in the sugar market, where only sugar coming under the above definition is sold as such.

In order that the essential features of this specific sugar may be readily grasped, it may be as well to briefly describe the principal points in the manufacture, especially those in which the divergence from the general lines of sugar-making occurs.

As doubtless your readers are aware, cane juice consists chiefly of a solution of sucrose, with small and varying proportions of glucose, albumenous pectinoid and mineral matters, etc., and with a colouring matter, *yellow* when free and *green* when united with bases, with which it forms feeble combinations.

As the cane juice comes from the mill, it is treated with sulphurous anhydride, prepared by burning sulphur in a current of air. Distinct acidity is thus imparted to the juice, and the colour of this, which, as it came from the mill, was dirty yellow, is now changed to a delicate greenish-yellow. This change of colour is due to the decomposition of the green colouring compound mentioned above, by the organic acids sat free by the sulphurous acid.

The juice is now raised to a temperature of from 212° F. to 225° F. in a closed vessel through which a constant stream of juice is passing, and on emerging is treated with milk of lime, to effect precipitation of the albuminous matter. The smallest quantity of lime that will give a juice sufficiently clarified for subsequent crystallisation is used, so that the colouring matter may be preserved as much as possible in its free—and yellow—state ; but unavoidably, to a greater or less extent, according to the purity of the juice, the colour of the clear product drawn off from the subsided precipitate is of too green a colour to yield a high-class yellow sugar. Indeed, should, at this stage of the manufacture, sufficient lime be added to make the juice distinctly alkaline, although a bright clarification would be obtained, the colouring matter would be too much acted upon for any subsequent process to rectify.

In the old method of manufacture, where the concentration of the thin juice from the clarifiers to the state of syrup ready for the vacuum pan was carried out under atmospheric pressure on the costly and destructive " copper wall " ; the acidity produced by the passing over this, with its high temperature and tumultuous treatment, rectified to some extent this lime effect on the colouring matter, especially if the concentration were carried far during this process ; but this end was only attained by great loss in manufacture from immersion, etc.

In the present method, evaporation in vacuo takes the place of the "copper wall," and as, during this stage, if properly conducted, no chemical change of any moment takes place, the colouring effect of the lime has to be nullified before the juice is subjected to concentration. This is done by the addition of phosphoric acid to the clear liquor from the clarifiers, and subsequent brief ebullition in what are known as "eliminators." This agent is added in sufficient quantity to liberate enough of the organic acids to recall the yellow colour which the juice possessed prior to the addition of the lime, and, at the same time, a considerable quantity of the lime, whose organic salts are prejudicial in sugar-making, is got rid of. Care, however, must be taken, and this applies equally to the "sulphuring" in the first stage of manufacture, that enough is not added to give rise to free phosphoric acid in the liquor, which would lead to loss later on.

Concentration of the liquor, which has received the finishing cleansing touches during its stay in the "eliminators," is now carried on in vacuo in some form of "multiple effect" until a density of from 25° to 35° Baumé is arrived at. A light, yellow-coloured syrup is thus obtained, which is immediately further concentrated and granulated in the vacuum pan.

When this operation is finished, a stiff magma of crystals and "mother liquor" or molasses, of a delicate greenish gold colour is obtained, and to further improve upon and fix, as it were, this colour, a solution of stanous chloride, in the proportion of about 1·3 lbs. of the latter to a ton of sugar is added. This magma or "massecuite" is now subjected to a separating process in the centrifugals, and the light yellow crystals thus obtained, the greater portion of the tin passing away in the molasses.

It is thus seen, and I wish to particularly impress this upon your readers, that the colouring matter of Demerara crystals is emphatically the colouring matter of the cane juice, and that the great object of the manufacture is to retain this colouring matter, and at the same time to produce a sugar of high saccharine richness.

And it is in doing this that another distinctive feature of this class of sugar is yielded, viz., the characteristic smell of cane juice which it possesses. It is owing to these qualities—colour, smell, and richness—that Demerara sugar holds the position it does in the sugar-making world.

A high-class yellow Demerara sugar, therefore, necessitates the use of a minimum quantity of lime, and the maintenance of a high degree of acidity throughout the process. I leave it to those practically acquainted with the working of cane juice to realise the difficulties associated with satisfactory manipulation on these lines, especially with the immature juice frequently met with in Demerara. Suffice it to say that, by efficient machinery and by careful and thoughtful work, it is possible to obtain results as regards the quantity of sugar extracted from the juice as good as those derived from the easier paths of the manufacture of refining crystals.

As already stated, these crystals occupy a high position among sugars in the home markets, and the result of this has been the production, by several refiners here, of distinct and avowed imitations, which, although sold in the market as "refiners' yellow crystals," are eagerly bought up by the trade and handed over to the consumer as "Demerara yellow crystals."

To produce these imitations, all that the refiner has to do is to purchase refining beet sugar, re-crystallise it, if necessary, so as to produce the crystalline condition of the Demerara sugar, and colour it, as it is admitted that it *is* coloured, by a preparation of a yellow aniline product, which possesses an aromatic odour, and which thus masks, when fresh, the characteristic unpleasant smell of beet sugars.

There is thus obtained, instead of a cane sugar possessing the actual colouring matter and smell of cane juice, a product prepared of inferior material and artificially coloured so as to simulate the real article.

This fraudulent substitution of an imitation for a real article goes on to an enormous and increasing extent. *Cœteris paribus*, it is bought for less in the market and retailed as the higher priced and genuine product.

The present position thus is, that the West Indian proprietors, after investing enormous capital in the form of machinery and skilled labour, in order to produce a pure and attractive sugar, find themselves compelled to undergo competition in the very market in which it would be thought their rights would be recognised, with adversaries who are allowed to carry on a trade in an imitation and inferior article with impunity.

There is one point in connection with the manufacture of Demerara sugar which may need comment, and that is, the use of a tin salt in the penultimate stage of the process. The use of this agent was commenced in 1881, but it was not until the following year generally adopted, on account of the uncertainty, from want of evidence, of the physiological position of small quantities of tin, the West India Committee having, in the meantime, investigated the question carefully, and satisfied themselves, by the evidence of Dr. Stevenson, Messrs. Hehner, Wigner, and others, that no fear need be entertained that a prejudicial influence on health would arise from the small quantities—from ·25 grains to ·33 grains to the lb.—of this metal found to be present in the sugars thus made. Dr. Stevenson, in experiments on dogs, carried out at the instance of the Committee, found that, with a diet almost entirely composed of the sugar in question, no interference whatever with health had resulted. Since then, some 500,000 tons have been consumed in the United Kingdom, without a single ill effect having been traced to its use.

Apologising for trespassing so far on your valuable space,

I am, Sir, faithfully yours,

Frederic J. Scard,

Chemist-in-chief to the Colonial Company, Limited.

16, Leadenhall Street, E.C.,

THE ANALYST.

NOVEMBER, 1890.

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A RAPID METHOD FOR THE ESTIMATION OF UREA IN URINE.

BY C. J. H. WARDEN, PROF. CHEMISTRY, MEDICAL COLLEGE, CALCUTTA.

As Sutton has remarked, the sodic hypobromite method of estimating urea in urine has given rise to endless forms of apparatus, the principle of construction being in all similar. In all the instruments hitherto proposed, with the exception, I believe, of Lunge's nitrometer, when it is used for the estimation of urea, the decomposition of the urea by the hypobromite has been accomplished in one apparatus, and the evolved gas measured in a separate tube. In the method now described the evolved nitrogen is measured in the same tube used for the decomposition of the urea; and the procedure is consequently much simplified.

The apparatus used for the decomposition of the urea and measurement of the liberated nitrogen, consists of a slightly modified form of Crum's well-known nitrometer. Crum's tube, as usually constructed, has a total length of 210 mm., an internal diameter of 15 mm., and a capacity of about 25 c.c., the cup at the top having a capacity of a little over 2·5 c.c. The modifications desirable in adapting the tube for the estimation of urea may be thus summarised:

(A) Trebling the length of the tube, and thus making it 630 mm. long. The capacity being increased to 75 c.c.; while the internal diameter is not altered.

(B) Grinding on to the lower open end of the tube a glass stopper on which ten narrow groves have been filed.

(C) Increasing the capacity of the small cup at the top of the tube to 5 c.c., and leaving it accurately graduated to hold 2·5 c.c. when filled to a certain mark.

(D) Using somewhat thinner glass than that employed in the construction of Crum's tubes, as no mercury is employed in the process.

It was found by Russell and West that 5 c.c. of a 2 per cent. solution of pure urea evolved 37·1 c.c. of nitrogen, and this volume was taken as the basis of graduation of the measuring tube. In the process now being described only 2·5 c.c. of urine are used, and as 2·5 c.c. of a 2 per cent. urea solution evolve 18·55 c.c. of nitrogen, this volume of gas will necessarily be equivalent to 2 per cent. of urea. One per cent. of urea is therefore equal to 9·27 c.c. of nitrogen, and the 9·27 c.c. volume is divided into ten equal parts, each part being equal to 1 per cent. of urea. These divisions are further subdivided into two, each representing ·05 per cent of urea. The graduations should be continued so as to indicate up to 3 per cent. of urea. All calculations are thus avoided, the observed volume of gas at once indicating the percentage of urea in the specimen of urine under examination.

In using the apparatus the following solutions are required:

1. A cold aqueous saturated solution of common salt.
2. Hypobromite solution made by dissolving 100 grains caustic soda in 750 c.c. of distilled water and adding 25 c.c. of bromine. This solution should be freshly prepared in small quantities as required for use.

To use the apparatus, the stop-cock should be first slightly greased and closed, the tube inverted, and the ground stopper removed. The hypobromite solution is then poured into the tube to overflowing, the stop-cock opened to allow a drop or two to escape and thus displace the small column of air in the bore of the stop-cock. The grooved stopper is then pressed home, the excess of hypobromite solution escaping by the grooves in the side of the stopper, in the same manner as the superfluous fluid escapes through the capillary orifice in the stopper of a specific gravity bottle. In this way the tube is filled with the hypobromite solution, all air being excluded. The tube is now inverted, held under a tap of water, and the cup, etc., thoroughly washed and then most carefully dried.

The tube is now stood in a small trough of urine—a finger-bowl answers very well—and supported by a filter-ring or clip. The sample of urine to be examined is then poured into the dry cup up to 2·5 c.c. graduation, any excess of urine accidentally introduced being removed by a fine fold of blotting paper. The cup is then filled up with brine, which is well mixed with the urine by a small and slender glass rod. The object of diluting the urine with brine is to increase its density. If ordinary urine were introduced into the tube it would float upon the surface of the hypobromite and only a slight reaction would immediately occur at the point of junction of the two fluids, but by increasing the specific gravity of the urine in the way indicated, it flows through the hypobromite solution, and brisk effervescence ensues. If the urine solution were allowed to enter the tube while closed with the grooved stopper, a portion of the evolved gas would escape through the bore in the stop-cock. In order, therefore, to avoid loss of gas from this cause, the grooved stopper is first removed before urine is allowed to escape from the cup into the tube. In allowing the urine to enter the tube, the stop-cock should be opened sharply and a small amount of urine in a full stream allowed to enter; decomposition of the urea at once ensues, and the gas collects at the top of the tube. When effervescence has ceased, more urine is introduced, and so on, until the cup has been emptied. The cup is then filled with 2·5 of dilute brine, and rinsed round by means of the glass rod; the whole of this brine is then allowed to enter the tube at one operation. The same amount of diluted brine is then again placed in the cup, rinsed round, and allowed to enter the tube. The tube is now grasped by the right hand, the thumb being tightly pressed against the open end, and the contents thoroughly agitated.

Lastly the tube is placed in a vessel of water, and the thumb removed, when the saline solution flows out, and water occupies its place. If, while the open end of the tube is below the surface of water, water from a tap be allowed to flow down the tube, the temperature of the gas will be rapidly reduced to that of the water. The remaining operation consists in measuring the volume of gas with the usual precautions, such volume indicating the percentage of urea without the trouble of any calculation. Corrections for temperature and pressure can, of course, be applied if necessary.

In using the tube, if the evolved gas should exceed the volume yielded by 3 per cent. of urea, the urine must first be diluted with an equal volume of water, and 2·5 c.c. of the diluted urine, and in the manner already described; the percentage of urea as indicated by the volume of gas being doubled.

The points to which special attention may be drawn in connection with this method of estimating urea, are—

1. Extreme simplicity of apparatus; all the various operations, of measuring the urine, decomposing the urea, and measuring the volume of evolved gas, being performed with one instrument.
2. Ease and rapidity of manipulation.
3. Results as accurate as those afforded by any of the various modifications of Russell and West's original method.

Messrs. Cetti and Sons, of Brooke Street, Holborn, are prepared to supply these modified Crum tubes, with a guarantee that the graduations are accurate.

ESTIMATION OF FUSEL-OIL IN SPIRITS.

By A. Stutzer and O. Reitmair.

(*Continued from page* 193.)

The Concentration of the Fusel by Fractional Distillation.

The possibility of removing the fusel from strong spirits by fractional distillation has often been talked about. Whether portions absolutely free from fusel could thus be obtained was not really proved, because there was no known reliable method to positively detect the presence of amylic alcohol in spirits of wine. An old test consisted in the oxidation of the fusel to valeric acid and analysing one of its salts. The process of Marquard, consisting in shaking first with chloroform, and then washing this with water, does not give good quantitative results. However, our chloroform process enables us to detect very small differences in fusel, and has been very useful in determining the value of the fractional distillation.

It is quite certain that when applying the process to very strong alcohols, products are at last obtained which cannot be further purified. We must, therefore, consider these fractions free from fusel as long as we do not succeed in proving its presence by other means, and ought only to use such a product as a check spirit in our process. For the carrying out of the following experiments, however, it was, as will be easily understood, not necessary to use such a highly purified spirit, but use was made of our spirit C.

Mixtures were made which contained respectively, ·1, ·05, and ·02 per cent. of amylic alcohol. In each case 1,000 c.c. of the sample were distilled, and 100 c.c. separately collected. The boiling points of the first nine fractions were constant between 78·5° and 78·9° C.

AMOUNT OF AMYLIC ALCOHOL BY VOLUME IN ORIGINAL ALCOHOLS.

	A	B	C	D
	·10	·15	·02	None
Fraction.	V. 15 = c.c.	V. 15 = c.c.	V. 15 = c.c.	V. 15 = c.c.
1	54·80	——	——	54·82
5	54·56	——	——	——
7	54·51	——	——	——
8	54·53	——	——	——
9	54·50	54·52	54·51	54·50
10	55·74	55·09	54·82	54.72

The difference between V. 15 of the ninth and tenth fraction amounts to

1·24 ·57 ·31 ·22

According to the former analysis, an increase of ·01 c.c. must be considered equivalent to ·0028 c.c. of amylic alcohol. Therefore A contains ·347, B ·161, C ·087, and D ·062 per cent. As, however, the distillates had first to be diluted up to 320 c.c. before applying the process, the true percentage of amylic alcohol becomes

A 1·1104, B ·5152, C ·2784, D ·1984.

If we now deduct the amount found in D we get

A ·912, B ·3168, C ·08 c.c.

The bulk of the fusel is therefore found in the last fraction, and if the spirit is very poor in alcohol it is advisable not only to distil one, but even two or three litres of the sample and operating on the last 100 c.c.

To answer the question in how far the distillation process succeeds when the spirit is not very strong, and in how far dehydraters influence the result, the experiment (A) was repeated, only with this difference, that the mixture was first mixed with 200 c.c. of water, and the alcoholic strength thereby reduced to 80 per cent. (experiment E). In another experiment (F) this same spirit was first mixed with 50 grams. of calcined potash, and in a third experiment (G) with 150 grams. of this substance. The several fractions showed the following boiling points:—

	E	F	G
	80 Per Cent. Spirit without Potash.	Same with 50 grms. Potash.	Same with 150 grms. Potash.
1	79·3°	78·8°	78·6°
2	79·4°	79·0°	78·6°
3	79·6°	79·0°	78·6°
4	79·8°	79·1°	78·6°
5	79·9°	79·2°	78·6°
6	80·0°	79·3°	78·6°
7	80·1°	79·4°	78·6°
8	80·2°	79·5°	78·6°
9	80·7°	79·7°	78·6°
10	81·2°	79·9°	78·7°

The separate fractions of G contained from 95 to 96 per cent. of pure alcohol, and the last fraction measured about 100 c.c. The distillates of F had a smaller percentage of alcohol, and those of E were still weaker. Therefore from E and F another 11th fraction was collected, boiling from 81·3° to 80·2° C.

The chloroform shaking gave the following results:—

Fraction.	E	F	G
	c.c.	c.c.	c.c.
9	54·63	54·55	54·51
10	54·64	54·69	55·76
11	55·13	55·71	——

The volumes of the last fractions of the spirits after proper dilution were—

E 320 c.c. F 260 c.c. G 310 c.c.

If we now deduct the increase of the chloroform of A we get

E ·41 c.c. F ·99 c.c. G 1·02 c.c.

which corresponds with ·1148, ·2772 and ·2856 per cent. of amylic alcohol in a 30 per cent. spirit.

The quantity of amylic alcohol in the last fraction is therefore—

E ·368 F ·721 G ·885 c.c.

and the loss in fusel—

·632 ·279 ·115

or calculated on 80 per cent. spirit—

·053 ·023 ·01

It is therefore plain that on using a dehydrater, even a weaker spirit may be made to retain the bulk of its fusel till the last fraction. It is, however, necessary to use a large excess of potash, which quantity must, of course, be regulated by the amount of alcohol the sample contains. As the greater part of the fusel remains in the retort, it is necessary to finally dissolve the potash in water, and then to distil another 100 c.c. The liquid having a high boiling point, all amylic alcohol passes out in the distillate which is then used to dilute the last alcoholic fraction. In the beginning of the distillation we use a brine bath, but at the last the naked flame, or else a paraffin bath. The loss in amylic alcohol, which must be expected from the use of potash, is only ·01 per cent. of the whole amount present. W. Fresenius says the smell, either before or after fractioning of the various distillates, is qualitatively a better proof for the presence or absence of fusel than the chemical or physical processes, but this view is not confirmed by our experiments. We found, in fact, that whenever fusel can be detected by the smell, it is quite possible to quantitatively determine it, but if the amount is supposed to be very small, we use, as already stated, not one litre, but even two or three litres of the sample for the fractional distillation.

Our Experiences on the Fractional Distillation of Spirits Containing much Fusel.

If the sample contains so much fusel that it may be safely detected without a fractional distillation, the experimental error, increased by the necessary dilution, amounts to about ·06 per cent. Analysts will have to be content with this degree of accuracy when analysing very bad spirits. The separation of the sample in fractions free from fusel, and those richer in it, then becomes more and more impossible. When distilling one litre of alcohol with ·05 per cent. of fusel, only the last 50 c.c. are contaminated; with ·1 per cent. the last 100 c.c., with ·15 per cent. the last 300 c.c., and with ·2 per cent. the last 500 c.c. And if the amount of fusel reaches ·5 per cent. the first 300 c.c. of the distillate are already contaminated.

It will now be understood why it is so difficult to purify spirits on the larger scale, as this can only be done by the most improved distilling apparatus and aid of chemical means. The process of Traube, shaking with saline solutions, such as ammonium sulphate, by which a concentration of the fusel is obtained, is only then of any use when very large quantities of the salt are used. For instance, to concentrate the fusel from a litre of alcohol containing 1 gram. of amylic alcohol into 100 c.c., it was found necessary to shake with no less than 15 litres of a saturated solution of ammonium sulphate. The experiments were therefore not continued.

Testing of Various Samples of Purified Spirits.

Our process to increase the fusel by fractional distillation and then to estimate it quantitatively, is as yet only practicable in the case of spirits poor in fusel, for which it, however, possesses an undoubted advantage. From our experiments with pure spirits, and the same when mixed with a definite amount of fusel, one can see that intermediate fractions are obtained which give a constant chloroform increase. (We take it, of course, for granted a chloroform of the same quality is used.) Instead of only testing the last, or the two last fractions, it is always as well to test one of the earlier ones so as to have a better check on the analysis.

No. 1. *Sample.*—First-class potato spirit, *so-called* spirits of wine, from the later fractions of a distillation, containing 95·5 per cent. of alcohol. 1,000 c.c. were allowed to stand on 100 grams. of dry potash for two or three hours at the ordinary temperature and then fractioned. The boiling point remained constant at 78·7° until the last drops distilled over. The remaining potash was dissolved in water and 100 c.c. of this distilled off.

Last fraction from 100 c.c.—
Noticed volume V = 54·55 T = 14·9° C.
Volume at normal temperature V 15 = 54·56
Last but one fraction from 100 c.c. V = 54·50 T = 14·8[*]
V 15 = 54·52

The increase in the chloroform volume is for both fractions a little less than with the spirit sent as pure. On the other hand the intermediate fraction of the last-named give a chloroform increase of ·06, and its ninth fraction an increase of ·02 c.c. As an increase of ·01 c.c. means ·0028 c.c. of amylic alcohol, ·02 c.c., ·0056 per cent. Allowing for the dilution up to 320 c.c., we get ·01792 c.c., = ·0017 per cent. by volume of amylic alcohol, a quantity so small that it may be put to experimental error and be disregarded.

No. 2.—A strong spirit, prepared by a new process from maize containing 95·65 per cent. of alcohol.

1,000 c.c. were distilled with 100 grammes of dry potash. The boiling point kept constant at 78·8° C. until the last fraction. The treatment was the same as in No. 1. The last fraction of 100 c.c. V = 54·55. Consequently the spirit must be considered free from fusel. The first fraction had a faint ethereal smell. On testing this first fraction V 15 = 54·69.

The shaking out of the spirit without previous distillation gave

V 15 = 54·71.

After rectifying over KHO we got

V 15 = 54·48
V 15 = 54·51

showing here the necessity of rectifying over caustic potash.

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[*] Using the same chloroform in the small Hersfeld's apparatus V 15 = 20·80 c.c.

No. 3.—Rectified corn brandy contained 95·2 per cent. of alcohol. 1,000 c.c. were distilled with 100 grams. of potash. The boiling point kept constantly at 78·7° C., and only reached 79·2° at the end of the distillation.

Last fraction 100 c.c. V = 54·49 ; T = 14·9
Corrected V 15 = 54·50
V = 54·51 ; T = 15·1
Corrected V 15 = 54·50.
100 c.c. of the last but one fraction V 15 = 54·50.

The spirit is to be regarded as absolutely free from fusel, but did not, however, possess such a pure odour as the first, and gave after dilution, but without previous distillation,

V = 54·71 ;

and after rectifying over caustic potash

V = 54·69.

So it seems this sample contained volatile impurities which were not removed by KHO.

The first portions of the distillate gave respectively

1.—V 15 = 54·80
2.—V 15 = 54·72.

From these figures it is plain that the testing of the last fractions is the best plan in any case ; better than testing the whole of the spirit, even after rectifying with caustic potash.

It seems that spirits often contain volatile impurities which interfere with the analysis even when the sample has been rectified over potash or caustic potash. These bodies will certainly cause a too high result with spirits, which, being rich in fusel, cannot first be distilled, but it is a question whether this error is at all considerable. Further experiments will have to decide this.

EXPERIMENTS WITH TRAUBE'S CAPILLARIMETER.

Some time ago we have, like Sell and Mayrhofer, pointed out that the estimation of fusel in brandies by this instrument is certainly easy of execution, but that the process was inferior in accuracy to the shaking-out process. At the same time as we did the other experiments, we carried out some more experiments with the capillarimeter, as the spirits used could not contain any large quantity of ethereal oils. We used the instrument as described by us in the *Repert Anal Chemie*, 1886, p. 386, with micrometric screw, and first determined the radius of the capillaries with the aid of quicksilver. A measured column of the mercury was then weighed, and so the volume calculated. The radius was

$$z = \sqrt{\frac{v}{\pi . h}} = \cdot 1793$$

The height increase for 20 per cent. alcohol was 49·5 mm. at 15° C. in our apparatus. If temperature was over 15° C., ·18 mm. had to be added for each degree. The instructions accompanying the apparatus were not correct, and it is, therefore, no matter of surprise that not much confidence is felt in the method. The figure for water is 83·5 at 20·2° C. Pure amylic alcohol was now mixed with alcohol and water in such proportions that the liquid contained exactly 1 per cent. of fusel, and the specific quantity of the same exactly corresponded with a 20 per cent. ethylic alcohol. By diluting with a 20 per cent. pure alcohol, mixtures were obtained which contained from ·1 up to 1 per cent. of fusel. The figure of each mixture was then determined. It amounted on an average to ·78 mm. for every ·1 per cent. of fusel, the increase being remarkably constant, and any slight differences were most likely due to experimental error. We, however, think that if the amount of fusel is large, the figure gets slightly less and believe it to be safer to take the figure as ·80 when the quantity of fusel lies between ·1 and ·5, and to reckon ·76 when between ·5 and 1·0 per cent. This difference of ·04 mm. has really no practical importance, as it is not possible to read off to less than ·1 mm. In using the capillarimeter it must be noticed that the alcohol must not be allowed to stand in the open basin or beaker for hours, but that the reading ought to be made after say five minutes. Alcohol being volatile, its strength in the open dish rapidly alters, particularly in a warm room.

We have performed a great number of estimations of fusel, and agree that the apparatus, on account of its simplicity and convenience, is in many cases an excellent one for the sorting of spirits, but we stick to our previous opinion that when great accuracy is required, the shaking process is much safer and more accurate.

It would not be interesting to give here our numerous figures of the height increase, and will content ourselves with mentioning some more results of our fusel-concentrating process.

1.—·5 c.c. of amylic alcohol were mixed with a 1,000 c.c. of a 96 per cent. spirit, and then fractionally distilled after addition of 100 grams. of potash. The last fraction, when made up to 20 per cent. strength, measured 482 c.c. The height increase compared with pure 20 per cent. alcohol was ·75 mm., corresponding with ·094 per cent. by volume of fusel, or ·453 per cent. in 482 c.c. liquid. As the original mixture contained ·5 c.c., or ·05 per cent. of fusel, the loss during the distillation must have amounted to ·0047 per cent.

2.—1·0 c.c. of amylic alcohol was mixed with 1,000 c.c. alcohol and 100 grams. of potash, and then fractioned. On testing the last in 100 c.c., ·912 fusel was found, showing a loss of ·0088 gram. or ·088 per cent. by volume.

3.—1·5 c.c. amylic alcohol + 1,000 c.c. spirit + 100 grams. of potash were mixed and fractioned—

Last fraction	·888
„ but one	·185
„ „ two	·185
	1·258

Therefore ·242 gram. of fusel oil, or ·0242 per cent. by volume, had past over with the first portions, and this confirms once more our idea that the process is only useful when the amount of fusel does not exceed ·1 per cent.

The numbers just mentioned still want a correction. To dilute the fusel the same spirit was used as in the experiments with the shaking apparatus. This spirit was not altogether free from fusel, but contained (by that process) ·19 c.c. fusel oil. To get approximately correct results, we must, of course, allow for this, and then we get the following figures:—

1 litre of spirit was mixed with	Recovered in shaking apparatus.	Recovered by capillarimeter.
·5 c.c. fusel; last fractions	·31 c.c.	·26 c.c.
1·0 c.c. " " "	·91 c.c.	·72 c.c.
1·5 c.c. " 3 last fractions	—	1·06 c.c.

THE DETAILS OF OUR PRESENT PROCESS.

The estimation of the fusel by shaking with the chloroform must first of all be done in the ordinary way after distilling the spirit with caustic potash, in the fluid diluted up to 30 per cent. If the analysis should show a result exceeding ·15, the analyst must take this as practically accurate. If, however, less than ·15 per cent. is found, 1,000 c.c. of the spirit and 100 grams. (or more) of calcined potash are introduced into a large fraction apparatus, and after standing for a few hours it is distilled off. The first 500 c.c. may be collected together, but after this every 100 c.c. are separately collected. After everything is distilled over, the retort is allowed to cool, and 250 c.c. of water are introduced. 100 c.c. are now distilled off from a paraffin bath, and the distillate united with the last alcoholic fraction. Now every fraction must be diluted up to 30 per cent. strength and (the last one first) examined in the apparatus.

The dilution up to 30 per cent. by volume must be done with the greatest possible accuracy; at all events, the experimental error should not exceed ·1 per cent., which will already cause a chloroform increase of ·03 c.c. The best shaking apparatus is such a one which has room for 250 c.c. of the spirit and 50 c.c. of chloroform. The central narrow tube of the apparatus should hold 50—56 c.c., and be divided into ·05 of a c.c., and one should be able to judge ·01 of a c.c. The temperature during the measuring of the fluids and the reading off should be between 14·5—15·5° C. The correction for temperature is made by allowing ·01 c.c. for every .1° C. either adding or deducting the same.

To get truly comparable results in the analysis of different kinds of spirits, we recommend, not only to state the percentage of fusel as found, but also to calculate its proportion to the alcohol which the sample contains.

DETERMINATION OF LITHIA IN MINERAL WATERS.

By E. WALLER, PH.D.*

PRACTICALLY, three methods are now available. 1. The phosphate method (Mayer's modification) (*Ann. Chem. u. Pharm.* 98, 193). 2. The amylalcohol method (*Gooch*, *Am. Chem. Jour.*, 9, 33). 3. The fluoride method (*Carnot*, *Bull. Soc. Chim.* [3] 1, 280).

Rammelsberg's method (*Pogg. Ann.*, 66, 79), somewhat similar in principle to that of Gooch, in that it depends upon the comparatively greater solubility of lithium chloride in an organic solvent, has been comparatively little used, on account of the difficulty and expense involved in obtaining the pure anhydrous alcohol and ether necessary for the process. Moreover the experiments of J. L. Smith (*Am. Jour. Sci.* [2] 16, 56), rearranged in convenient form for reference by Gooch do not indicate that it is very satisfactory in its application, even with the best of care. Some indirect processes, such as the weighing of mixed chlorides of sodium, potassium and lithium, and then determining the chlorine and potassium (Bunsen, *Ann. Chem. u. Pharm.*, 122, 348), have been also proposed, but they are troublesome in execution, and likely to be unsatisfactory in result.

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* Journal of the American Chemical Society.

For all of these processes, it is necessary to obtain from some known quantity of the water, the alkalies as chlorides free from admixture with other bases, and in most cases, a considerable proportion of the sodium and potassium salts, which usually predominate largely over those of lithium, must be removed. To accomplish this the usual method may be followed, acidification with hydrochloric acid, evaporation, treatment with barium hydrate solution, removal of the excess of baryta by ammonium carbonate, driving off the ammonium salts, and extraction with alcohol or alcohol and ether, to take out the lithium chloride which is inevitably accompanied by some sodium and potassium chlorides. Throughout this treatment, the spectroscope must constantly be used to determine when the extraction or washing is complete, and these preliminary operations often prove very tedious. Some suggestions in this connection may be of value. The small platinum wires used to test the precipitates, solutions, etc., need critical examination. A wire which has been once used with lithium salts may perhaps be held in the flame until it will give no trace of colour to the flame, nor show the lithium line by the spectroscope, but on moistening with hydrochloric acid and inserting in the flame, the line will show almost as brightly and distinctly as if no lithium had been removed from it. Repeated scouring, immersion in acid and insertion in the flame, or long soaking in acid may be necessary to remove this trace of lithium from the wires. It has been found convenient to keep several wires dipping into a test tube partially filled with dilute hydrochloric acid, and to use them in succession, so that each wire shall have a tolerably long immersion in the acid, before being tested again, as a preliminary to using it for a test on a precipitate, etc.

Barium precipitates ($BaCO_3$ and $BaSO_4$) when formed in the presence of lithium compounds, carry down and retain perceptible quantities of lithia (by spectroscope test) with great persistency. The well-known tendency of the barium sulphate to drag other salts with it *seems* to be greater in the case of lithia than in that of other alkaline salts, although this may perhaps be due to the exceeding delicacy of the spectroscopic reaction. Precipitation in a rather dilute solution, and rather liberal washing is usually the most convenient course to pursue in the case of the precipitation of $BaSO_4$ in presence of lithium salts. With $BaCO_3$, re-solution in HCl, and re-precipitation with ammonia and ammonium carbonate is most effective. If the proportion of lithium is large, re-solution and re-precipitation a third time may be advisable. The precipitate produced by barium hydrate, unless consisting largely of sulphate, does not give so much difficulty in the washing out of the lithia, except when it has been exposed for some time to the air of the laboratory containing CO_2.

A word further as to the decomposition of LiCl by heat. Direct quantitative estimations upon the subject were not made in this investigation, but the phenomena noted tend to confirm Mayer's remark that under the influence of heat in presence of water, lithium chloride has a tendency to exchange chlorine for oxygen.

A solution containing lithium chloride is evaporated to dryness with difficulty when placed on the water bath, and if it finally is made to *appear* dry after prolonged treatment in this manner, more of the material is slow to re-dissolve in water (apparently because of the formation of lithium hydrate) than if the same solution is evaporated nearly to dryness and the moisture driven out by careful ignition over a naked flame.

Assuming in every case that one has obtained a concentrated aqueous solution from a known quantity of the water, containing all of the lithium and some of the potassium and sodium as chlorides, but no other bases, the phosphate method would be as follows:—

Add an excess of hydro-disodium phosphate, and then a moderate excess of pure sodium hydrate, evaporate to dryness, re-dissolve in water by the aid of a gentle heat, add an equal volume of strong ammonia, digest warm for some time, allow to stand for twelve hours, filter and wash with a mixture of equal volumes of ammonia and water, and finally ignite and weigh as Li_3PO_4. A second or third portion of precipitate may be recovered, by evaporating the filtrate and washings, adding ammonia and allowing to stand as before, filtering, etc. The chief difficulty with the accuracy of the process, consists in the practical impossibility of obtaining all the lithium as phosphate, free from any other alkaline salts. Too much washing will cause appreciable amounts of lithium phosphate to go into solution. Indeed, in my experience the filtrate and washings have always showed a decided lithia line in the spectroscope, from the start. On the other hand, too little washing leaves some alkaline salt along with the lithium phosphate—shown by its tendency to cake on ignition—but whether it cakes or not, lithium phosphate separated by this method when tested by the flame, almost invariably gives so strong a sodium flame, as practically to obscure the red of the lithium to the naked eye. Consequently it becomes to a considerable extent a matter of judgment, to decide when the washing is completed, and *then* the amount of lithium phosphate obtained is a compromise between the precipitate dissolved off by washing and alkaline salts left with it. However, by the aid of a little experience, the error can be usually brought within moderate limits, if the proportion of water which the chlorides represent is sufficiently large (ordinarily 10 to 20 litres). The use of such large quantities of water is naturally attended with more or less labour, in evaporation, removal of bases, etc., and is in itself objectionable aside from the sources of error inherent in the method of determination. This method has, however, until recently been practically the only one in general use.

The method of Gooch used by him in the examination of the waters of the Yellowstone Park (*Bull. U. S. Geol. Survey*, No. 47, also *Chem. News*, 59, 113, *et seq.*), may be described as follows:—

The concentrated solution of the alkaline chlorides should contain only about 0·2 gram. of salts in all. To this solution, in a casserole or dish, is added 30 to 50 c.c. of pure *anhydrous* amyl alcohol. The vessel is then heated on a sand bath over a low flame, so as to boil off the water through the amyl alcohol, leaving the undissolved salts adhering to the sides of the dish. The heat is kept up until the volume of amyl alcohol has been reduced to about 15 or 18 c.c. after cooling. A few drops of hydrochloric acid are added to restore to the form of chloride any lithium oxide or hydrate which may have been formed, and the heating is repeated for a short time. The amyl alcohol is then filtered through paper, or through a Gooch crucible, into a measuring cylinder, and its volume noted (usually 10 to 15 c.c.). In case the proportion of lithium is large the undissolved salts should be taken up with a little water, and the treatment repeated in the same way as just described, the amount of amyl alcohol which has been heated with the chlorides being measured as before. The salts are then washed with cold amyl alcohol until no trace of lithium is perceptible in them by the spectroscope; the filtrate and washings are evaporated in a weighed platinum dish, and the chlorides converted into sulphates, ignited and weighed. From this weight, for every 10 c.c. of amyl alcohol which remained in contact with the chlorides after heating, the following deduction is made:—

When only sodium and lithium chlorides were present,	0·00050 gram.
„ „ potassium „ „ „ „	0·00059 „
When both sodium and potassium, as well as lithium chlorides were present	0·00109 „

The cold amyl alcohol used for washing dissolves so little that it is needless to take it into account.

The relative solubilities of NaCl, KCl, and LiCl in amyl alcohol, as determined by Gooch, are essentially:—

NaCl	1 in 30,000, or 0·0041 gram.	in 100 c.c.
KCl	1 „ 24,000, or 0·0051 „	100 „
LiCl	1 „ 15, or 6·60 „	100 „

Temperature seems to have but little influence upon the solubility of NaCl and KCl.

Pure amyl alcohol freed from water by boiling, if necessary, is indispensable.

Gooch's test experiments with mixtures of pure salts may be here quoted, arranged in a form slightly different from that given in his paper. The Li_2SO_4 obtained was calculated back to LiCl in every case. The error is noticeably greater in the presence of potassium chloride.

Ex. pt. No.	Conditions.		Li Cl taken.	Errors in corrected weight of Li Cl found.
(23)	Na Cl only	Single	0·1298 grm.	0·0002— grm.
(24)	„ „	Extr'n	0·1227 „	0·0002— „
(32)	„ „	Double	0·1287 „	0·0007— „
(33)	„ „	Extr'n	0·1347 „	0·0006 + „
(26)	KCl only	Single	0·1256 „	0·0008— „
(27)	„ „	Extr'n	0·1287 „	0·0010— „
(34)	„ „	Double	0·1125 „	0·0003 + „
(35)	„ „	Extr'n	0·1251 „	0·00011 + „

The average of these errors is 0·0001— with a range from 0·0010— to 0·0011 + or if we calculate to the equivalent in $LiHCO_3$ from 0.0016— to 0·00176+, a difference of 0·00376 gram.

The process has the advantage that the sodium and potassium chlorides are left in a condition for the determination of those bases, in which case, however, an allowance must be made for the small amounts dissolved by the amyl alcohol which was heated with the chlorides. One disadvantage of the process is to be found in the fumes of the amyl alcohol which, even in a well ventilated laboratory, is a source of great discomfort to most analysts.

The small amount of water, 100 to 200 c.c. that can be used for this process is advantageous, though for those accustomed to the use of the phosphate process, the amount seems hardly large enough to give a fair average, and to average on a larger amount requires the concentration of the LiCl by extraction with alcohol or alcohol and ether. In some of the first trials made with the process, the amount of mixed chlorides experimented upon considerably exceeded 0·2 gram., probably nearly 1 gram., and in some cases more. This was because Prof. Gooch's paper seemed to imply that the operation might be reasonably expected to be successful, when applied to quantities ordinarily handled in analytical work. In the case of one water it did do so, but with another water, containing more lithia as well as alkaline salts, it was not. The removal of all the water in the manner described was a matter of extreme difficulty, and, curiously enough, a limit seemed to be reached, beyond which the LiCl was extracted, but slowly and with great difficulty. The results may prove interesting.

The alkaline salts from two equal quantities of a water, each lot amounting to between one and two grams., were treated as described :

Obtained from A	0·2400 grm. Li_2SO_4	from B	0·2354 Li_2SO_4
Found in insoluble part. f. A	0·0974 „	„ B	0·0943 „
Total	0·3374 „		0·3297 „

The control process used in this case was Carnot's fluoride method, which cannot, however, be regarded as absolutely free from imperfections. It is as follows : the mixed alkaline chlorides after, evaporation nearly to dryness, are extracted with a mixture of about equal volumes of alcohol (of 90 per cent. or over) and ether, so as to obtain the LiCl comparatively free from the others. It was found most convenient to add the alcohol ether mixture, and allow to stand for some time with frequent stirring, and then after standing over night to filter through a small filter, and wash with alcohol; one extraction will often suffice. A second extraction may, however, be necessary, the work being of course controlled by the indications of the spectroscope. After evaporating off the alcohol and ether, the salts are dissolved in the least possible quantity of water, and filtered into a weighed platinum dish. The filtrate and washings should then be concentrated to small bulk (5 or 10 c.c.) and pure ammonium fluoride and ammonia added ; after thorough mixing the dish is set aside over night for the LiF to precipitate. The solution is then decanted through a small filter, and the precipitate is washed by decantation three or four times (5 to 7 c.c. at a time), with a solution consisting of the reagent mixed with 5 to 10 times its bulk of ammonia ; between the decantations the solution must be allowed to stand some little time with stirring. The bulk of filtrate and washing (30 to 50 c.c.) is noted, the filter paper and contents placed in the dish, sulphuric acid added, and heat applied until the paper has been incinerated, and the lithium converted to sulphate, in which form it is weighed. To this weight is added 0·0040 gram. for every 7 c.c. of filtrate and washings, and the result estimated as Li_2SO_4 is calculated to Li, $LiHCO_3$, etc., according to the requirements of the case.

Care is necessary in preparing the reagent, and wash liquor.

Carnot seems to have found that the ammonium fluoride ordinarily supplied for laboratory use is the only member of the combination liable to contain impurities which would interfere (chiefly fluosilicic acid, which might precipitate alkaline fluosilicates), but experiments have shown that ammonia, which has been standing for some time in contact with glass, will give a cloud (presumably ammonium fluosilicate) with a mixture of solutions of ammonium fluoride and ammonia after boiling and filtering clear. This solution, so long as it contains a fair amount of free ammonia, appears to be without action upon glass. It has been found advisable therefore to make up (and cork up) the reagent and washing solution some time beforehand, and to filter off such portions as may be required at the time of using. Naturally, it is necessary to use for the final filtration, a filter paper which has been extracted with hydrofluoric acid. Schleicher and Schull's papers were found satisfactory in this connection. Carnot also recommends that the resulting Li_2SO_4 should be dissolved in 40 to 50 c.c. of water, and a test made for the presence of magnesium, which may have remained with the alkaline chlorides. If any is found to be present, it must be determined as phosphate and a correction made accordingly.

In connection with this process it was observed that lithium sulphate ignited in contact with the carbon of the filter paper, is especially prone to reduce to sulphide, and especial caution is necessary at this stage of the operation. The sulphide, when heated in contact with the platinum, attacks it in a very marked manner.

The process seems to be very good, although not rapid.

Its tendency is to yield results a little high, apparently because the allowance for solubility is usually larger than the actual amounts of precipitate dissolved. Test analysis tended to show also that unless the amounts of potassium and sodium chlorides present with the lithia are kept within narrow limits, the results will be high.

Unfortunately a number of the tests and comparison experiments with these methods have not yet been completed, and will have to be deferred to a second communication.

In order to test these methods upon water containing lithia, samples of several of the best known and widely advertised waters were purchased and submitted to examination.

The results were somewhat surprising, and shewed unquestionably that either the original analyses, on the strength of which those waters are now sold, were erroneous, on account of imperfection in the methods used, or, what is more probable, that the proportions of lithium in those waters are liable to great fluctuations.

The results given were chiefly obtained by Carnot's fluoride method, but were in several cases confirmed by the use of other methods. The most scrupulous care was exercised to be sure of obtaining *all* of the lithium in the waters under examination, the spectroscopic indications having been used at every stage of the process.

In the Farmville Lithia Water, purchased at the office of the company, no lithium could be detected by the spectroscope on moderate amounts of water. On evaporating eight litres of the water, and treating in the manner described for the concentration of the lithia into a solution of small bulk, a lithia line was obtained in the spectroscope, but the amount was found to be too small to admit of a quantitative estimation. The experiment was repeated with ten litres of the water, with essentially the same result.

With the Buffalo Lithia Water the reaction for lithium was more distinct, when considerable quantities of the water were concentrated. From twenty litres of the water was obtained lithium sulphate corresponding to 0·0185 part $LiHCO_3$ per 100,000.

In the Londonderry water, the lithia reaction could be obtained without great difficulty. Analysis of the water purchased by myself showed a little over four parts per 100,000. The company puts up some of the water in half-gallon bottles not charged with CO_2, and also some in pint bottles (called in their circulars "sulpho-carbonated"), which is charged with CO_2, and has also received the addition of some salts. The amounts of salts added appears to be somewhat irregular. For instance, the following results were obtained (results given in parts per 100,000):

	Total solids.	Loss on ign.
Londonderry, half-gallon bottles (aver.)	37·35	2·25
„ pint bottle, A	149·4	4·9
„ „ B	104·2	4·5
Average of eleven others	224·7	6·4

The variations in the eleven bottles were 221·3 to 231·4, for total solids. The proportion of lithium was essentially the same as for the still water.

I was told that several lots of water, purporting to come from these springs, had at times appeared on the market in which no lithia could be detected. As I learned that Dr. Endemann had obtained some water of that kind, I requested him to send me a bottle. He complied, and although the bottle bore all the labels and marks similar to those purchased by myself, no lithia could be detected in it. The water contained 5·2 parts total solids per 100,000. I have heard of others who had similar experiences.

I naturally desired to obtain samples of these water direct from the springs, taken by some one whom I knew to be disinterested. Attempts thus far have been unsuccessful. In the case of the Londonderry springs, all access is denied to visitors, and applications for water are referred to the bottling establishment in Nashua.

Of all the waters examined, purporting to be natural, the Saratoga Hathorn proved to be the strongest in lithia. The result of tests on this water are not at present in such form that they can here be recorded, but it suffices to say that the water contains fully as much as the analyses call for (twelve to fourteen parts $LiHCO_3$ per 100,000, corresponding to seven or eight grains per U. S. gallon).

Tests were also made on the waters manufactured and sold by Carl H. Schultz as containing lithia. They were found to contain a little more lithia than claimed. E. g., the formula on his "Vichy with Lithia," called for an amount corresponding to about fifty-seven parts $LiHCO_3$ per 100,000. The analyses showed sixty to sixty-two parts.

The results enumerated may be thus tabulated :

Designation of Water, etc.	Total Solids Per 100,000.	Loss on Ign.	Non-Volatile.	Use for Determination.	$LiHCO_3$ Per 100,000	Grs. U. S. Gal.	Grs. Imp. Gal.
Farmville Lithia, half-gal. bottles	16·4	1·1	15·3	8·L	traces.		
„ „	....	....	....	10·L	traces.		
Buffalo Lithia, half-gal. bottles..	93·2	6·5	88·7	20·L	0·0185	0·011	0·013
„ „	....	....	....	20·L	0·0135	0·008	0·009
Londonderry Litbia, h'lf-gal. bot's.	37·35	2·25	35·1	10·L	4·171	2·432	2·920
„ „	....	....	....	10·L	4·075	2·376	2·852
„ „	....	....	....	2·L	4·130	2·408	2·891
„ „ pts. (c'h'g'd CO_2)	....	....	....	2·L	4·129	2·407	2·890
„ „ „	....	....	....	4·L	4·074	2·376	2·851
„ „ (half-gal. Dr. E.)	5·1	1·	4·1	1·L		none	

THE USES OF HYDROGEN DIOXIDE IN QUANTITATIVE ANALYSIS, AND THE IMPORTANT METHODS FOR DETERMINING HYDROGEN DIOXIDE.

By C. H. Jones.*

The more important applications of hydrogen dioxide for quantitative purposes are given below.

Classen and Bauer † found that hydrogen dioxide can be used advantageously in the determination of hydrochloric, hydrobromic and hydriodic acids in the presence of sulphuretted hydrogen. The latter is oxidised to sulphuric acid, and the chlorine, bromine and iodine are then determined in the usual manner.

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* *American Chemical Journal.*

† Ber. d. deutsch. chem. Gesell. 16, 1061.

The oxidation of sulphur to sulphuric acid by hydrogen dioxide is also employed to separate arsenic, antimony, zinc, copper and cobalt from the sulphur in their sulphides.

The sulphur can be liberated as sulphuretted hydrogen and oxidised by hydrogen dioxide. They employed this method in the determination of antimony trisulphide, antimony pentasulphide, tin, cadmium, and iron sulphides. It was found that hydrogen dioxide will also readily oxidise sulphur dioxide. This reaction is therefore employed to oxidise barium and sodium sulphites and sodium hyposulphite to sulphates.

P. Ebell * determined lead dioxide by hydrogen dioxide in two ways. He used a known amount of the hydrogen dioxide, allowed it to decompose the lead dioxide, and then titrated the excess with potassium permanganate. He also effected the same decomposition, and measured the volume of the resulting gas. He recommends the former method. The latter method gave results which were too low as compared with those obtained by other methods.

G. Lunge † described a method for determining permanganate and manganese dioxide by means of an excess of hydrogen dioxide and sulphuric acid. The oxygen evolved was collected and measured, half coming from the manganese dioxide and half from the hydrogen dioxide.

The same author proposed later a method for determining the available chlorine in bleaching powder by means of an excess of hydrogen dioxide, half of the hydrogen evolved coming from the bleaching powder and half from the dioxide. But the volume of the oxygen set free is just equal to the total volume of the available chlorine in the bleaching powder.

A. Carnot ‡ determined chromic acid by hydrogen dioxide. The slightly acidulated bichromate solution was titrated with very dilute hydrogen dioxide, which was then standardised by means of pure bichromate. The end of the titration is shown by the absence of the blue colour when the dioxide is dropped into the chromic acid solution.

G. Lunge § recently recommended the slightly modified nitrometer for standardising potassium permanganate, and for determining bleaching powder and manganese dioxide by hydrogen dioxide and sulphuric acid.

Some of the above decompositions have been employed for the determination of the dioxide itself. These and other important methods for determining hydrogen dioxide will be briefly considered.

Barium dioxide was determined by Brodie || by treating it with hydrochloric or acetic acid in the presence of finely divided platinum or of animal charcoal, which decomposes the hydrogen dioxide catalytically. The apparatus, including the materials used, was weighed and the acid admitted to the barium dioxide. Decomposition having taken place, the apparatus was again weighed. The loss in weight equals the weight of the oxygen evolved.

In water solution, hydrogen dioxide has been determined by Schönbein. ¶ He acidified the solution with sulphuric acid in order to secure the complete reduction of the permanganate. The solution thus acidified was titrated with standard potassium permanganate until the first traces of colour appeared.

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* Rep. anal. Chem. 6, 141-43.

† Ber. d. deutsch. chem. Gesell, 18, 1872.

‡ Compt. rend. 107, 948.

§ Jour. Soc. Chem. Indst., January, 1890, 21.

|| Phil. Trans. 1850, 2, 779.

¶ Jour. für. prakt. Chem. 79, 78.

Another application of this same reaction has been proposed by F. Hamel.[*] He treated the dioxide with a permanganate solution, collected and measured the oxygen evolved. At the same time he standardised his permanganate solution for further use.

Instead of potassium permanganate, A. Riche [†] employed manganese dioxide and sulphuric acid for determining hydrogen dioxide. Tha oxygen liberated was collected and measured. Lead dioxide is also used for the same purpose.

A method has been proposed by A. Houzeau [‡] which is based on the following reaction in acid solution: $H_2O_2 + 2KI = 2KOH + I_2$. A measured amount of standard acid was introduced into the neutral solution of the hydrogen dioxide to be determined. A small excess of potassium iodide was then added. The iodine liberated was boiled off and the excess of acid titrated with standard alkali.

Bertrand [§] used the above reaction to determine barium dioxide and hydrogen dioxide. Instead of driving off the iodine and titrating the excess of acid, he titrated the liberated iodine with a standard solution of sodium hyposulphite.

For the detection and determination of small quantities of hydrogen dioxide, Schöne [‖] makes use of the decomposition of potassium iodide by the dioxide in neutral solution, and the detection of the free iodine by means of starch paste. He states that this method will detect 0·00008 gram. in a litre. When there is more dioxide present than 0·001 gram. in a litre, the method is not accurate, since the colour does not change in the same ratio as the substance. A milligram. and two-tenths to a litre give about the same colour as a gram. The minimum limit of accuracy is 0·0008 gram. and the maximum 0·001 gram. of the dioxide to the litre.

G. Lunge [¶] reversed his method for determining bleaching powder by hydrogen dioxide, and determined the dioxide by means of an excess of bleaching powder. The volume of the oxygen set free was measured, half coming from the bleaching powder, half from the dioxide.

Potassium bichromate has been used by Wilfart [**] for an approximate determination of hydrogen dioxide. The dioxide was treated with dichromate and sulphuric acid, and the liberated oxygen measured.

A. Carnot [††] titrated the dioxide with a standard solution of potassium bichromate in the presence of a little hydrochloric or sulphuric acid. Carnot states that this method is not as reliable as the permanganate method.

Attention is called to a piece of apparatus recommended by Maurice de Thierry [‡‡] for determining hydrogen dioxide.

Some work which I have done in this laboratory shows that the applications of hydrogen dioxide to quantitative analysis can be extended by the use of nitric acid instead of sulphuric. This works more satisfactorily where the formation of insoluble sulphates is involved. Thus, minium and lead peroxide yield thoroughly satisfactory results when treated with nitric acid and hydrogen peroxide. The commercial hydrogen dioxide should in all cases be diluted three or four times to prevent decomposition on standing.

For the determination of minium, an excess of the diluted dioxide and dilute nitric acid are placed in a 200 c.c. Erlenmeyer flask. About a gram. of minium is weighed in a weighing tube of convenient size, which is then placed upright in the flask. The flask is connected with a Hempel's burette or a Lunge nitrometer. The gas volume, after adjustment of pressure, is read. The weighing tube containing the minium is then over-turned into the mixture of nitric acid and hydrogen dioxide. In a few minutes the decomposition is complete. The increase in volume is ascertained and reduced to standard conditions of temperature and pressure. Half of the oxygen comes from the minium and half from the hydrogen dioxide. The water in the burette should be made slightly alkaline with sodium or potassium hydroxide, to remove any traces of carbon dioxide which may come from the action of nitric acid on impurities in the minium. The results obtained, expressed in percentages of oxygen were:

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[*] Compt. rend. 76, 1023.

[†] Jour. pharm. Chim. [6] 13, 249-56.

[‡] Compt. rend, 66, 44.

[§] Bull Soc. Chim. d. Paris 33, 148.

[‖] Zeit. für anal. Chem. 18, 154.

[¶] Ber. d. deutsch. chem. Ges. 19, 868.

[**] Zeits. für anal, Chem. 1888, 417.

[††] Compt. rend. 107, 948.

[‡‡] Jour. pharm. Chim. [5] 12, 501-2.

	Oxygen.
	2·40 per cent.
	2·39
	2·36
Average,	2·38 +

To test the above results, portions of the same specimen of minium were heated in a vacuum, the oxygen pumped away as rapidly as it was set free, and measured after removal of carbon dioxide. Under these conditions the minium passed completely into lead oxide. The results were:

	Oxygen.
	2·37 per cent.
	2·36
	2·36
Average,	2·36 +

We attempted to determine the oxygen in the same specimen of minium by heating it in hard glass tubes attached to a Hempel's burette and measuring the oxygen evolved. Platinum gauze was placed in the tube to facilitate the distribution of heat. The results, expressed in percentages of oxygen, were:

	Oxygen.
	2·19 per cent.
	2·21
	2·22
	2·26
	2·20
Average,	2·21 +

As will be seen, the results are too low. This is to be attributed to a partial recombination of the lead oxide and oxygen during cooling.

The method * sometimes employed for determining lead dioxide by heating in the air and ascertaining the loss in weight is subject to the same source of error.

The determination of lead dioxide was carried out in all respects like that of minium. The specimen should be finely pulverised, and a half-gram. or less used in a determination. The results with hydrogen peroxide and nitric acid, expressed in percentages of oxygen evolved, were:

	Oxygen.
	5·43 per cent.
	5·43
	5·42
Average,	5·42 +

Determinations of the same specimen by heating in a vacuum were carried out as in the former case. More carbon dioxide was found here than in the minium. ·3 per cent. of carbon dioxide was removed from the oxygen. The results, expressed in percentages of oxygen, were:

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[*] Zeits. für anal. Cnem. 14, 348.

Oxygen.
5·45 per cent.
5·43
Average, 5·44

The determination of manganese dioxide by hydrogen dioxide and sulphuric acid has been made by Lunge as above mentioned. But we find that nitric acid is preferable, as will appear below.

A specimen of manganese dioxide was ground and dried. Two determinations were made by Lunge's method, with the following results:

Oxygen.
13·13 per cent.
13·14
Average, 13·13 +

Two determinations of oxygen in the same specimen, made with the use of dilute nitric instead of sulphuric acid, gave:

Oxygen.
13·17 per cent.
13·24
Average, 13·20 +

The slightly higher results in the second case are, perhaps, due to the presence of ferrous iron in the ore, which are oxidised by the nitric acid, and loss in available oxygen avoided. To test this we added equal amounts of a ferrous salt to weighed portions of the manganese dioxide, and determined the oxygen by sulphuric and nitric acids respectively. The results obtained when sulphuric acid was used were slightly lower, while when nitric acid was employed they were practically constant. This tends to confirm the above idea.

That hydrogen dioxide can be determined by minium and nitric acid, or lead dioxide and nitric acid, will be seen from the following results. The specimen was first determined by calcium hypochlorite. The results are expressed in litres of oxygen obtained from one litre of hydrogen dioxide.

Results when calcium hypochlorite was used.

Oxygen.
5·890
5·890
5·880
Average, 5·8866 +

Results when minium and nitric acid were employed:

Oxygen.
5·900
5·910
5·910
Average, 5·9066 +

Results from the decomposition by means of lead dioxide and nitric acid:

Oxygen.
5·910
5·900
5·910
Average, 5·9066 +

Chemical Laboratory, John Hopkins University.

LAW NOTES.

SALICYLIC ACID.—IMPORTANT STATEMENT BY PROFESSOR CHARTERIS.

In the County Buildings, Glasgow, October 16th, David Reid, chemist, 156, New City Road, was charged before Sheriff Balfour, at the instance of Mr. Peter Fife, sanitary inspector, with having, on 3rd July, sold to Inspector Armstrong one ounce of salicylic acid, which contained 2½ per cent. of cresotic acid. He pleaded not guilty.

Mr. Angus Campbell, writer, who appeared for the respondent, said that what he sold was commercially known as salicylic acid, and was made under the pharmacopœia formula which was in general use. Cresotic acid was almost inseparable from salicylic acid, except under the formula recently adopted by Professor Charteris, who had discovered means of still further purifying the drug.

Mr. R. G. Ross, writer, who prosecuted, said that the object of the sanitary authorities was not to press for a penalty, or even for a conviction, but to let the public know what they were getting.

Professor Charteris, who was the first witness, said that he was professor of materia medica in Glasgow University. Salicylic was principally used for the treatment of acute rheumatism. It was taken inwardly. There were two kinds of salicylic acid—natural, which was obtained from salicin and oil of winter-green, and artificial, which was obtained from carbolic acid. He was at the Royal Infirmary in 1880, and used artificial salicylic acid for rheumatism. He found that when it was prescribed for two days in the ordinary dose it created bad symptoms, such as delirium, restlessness, and a desire to get out of bed. He tried natural salicylic acid, and it had no bad results. He then stopped the use of the artificial acid altogether, and since 1880 had used only the natural acid. He had known of serious results from the use of the artificial acid. During the summer of last year he made experimental researches, testing the natural and the artificial acid. The latter was a powdery substance like quinine, while the natural acid had clear, well-defined crystals, something like strychnine. He found that the artificial acid in ten-grain doses was fatal. He applied it to a rabbit weighing 2½ lbs., and it caused convulsions and death. He applied the same quantity of the natural acid to another rabbit, and there were no bad results. He experimented on the artificial acid with a view to taking out the impurities. The detection of the latter was mainly due to Mr. Henderson. The impurities were probably some form of impure salicylic acid and creosotic acid. The impurities taken out of the artificial acid had the appearance of a brown, dun powder. He made experiments with these impurities, and found that one grain of this brown powder was fatal to a rabbit weighing 2½ lbs. It was very simple to extract the impurities. Cresotic acid was poisonous. The impurities taken from the artificial salicylic acid were distinctly poisonous. His experience, fortified by that of other physicians, was that it was dangerous to the public to use artificial salicylic acid. In rheumatism it was necessary to give large and repeated doses, and these caused such symptoms as delirium and restlessness. These symptoms he accounted for by the impurities which were in the artificial salicylic acid. The natural acid cost four or five times the price of the artificial. Purified artificial salicylic acid cost 10d. and unpurified 9d. Kolbe's patent, by which the artificial acid was made, expired two years ago.

Cross-examined by Mr. Campbell: He scarcely thought that there were differences of opinion as to the result of using artificial salicylic acid. According to the pharmacopœia the melting point of salicylic acid was about 155 Centigrade. The article discovered by Kolbe was in almost constant use from the date of the patent in 1874 downwards. Kolbe's was the article known as artificial salicylic acid, and under the patent the discoverer had a monopoly of the manufacture. Since the expiry of patent chemists and druggists had, he presumed, dealt in the article manufactured under the Kolbe specification. The presence of 2½ per cent. of cresotic acid would not be a usual quantity, and there might probably be more. If "salicylic acid" were put in a prescription the chemist would supply the cheaper kind, but if witness wanted the other he would add the word "natural." The pharmacopœia recognised, but only vaguely, the natural acid as distinct from the artificial. When witness wanted the natural acid he always expressly said so, but he did not know if that was universally done. According to the formula in use down to witness's discovery he did not think that artificial salicylic acid could be got without the impurities. The natural article was made from salicin or else from oil of wintergreen. For one ounce of the natural acid forty or fifty ounces of the artificial were sold.

Dr. John Clark, City Analyst, stated that he examined the sample of acid bought from Mr. Reid, and found it to contain 2½ per cent. of cresotic acid. The melting point of the pure acid was 156·8, and the pharmacopœia gave it as about 155. The melting point of the sample was 154·7, and that was an indication of impurity. It had been assumed in this case that all the artificial salicylic acid produced under Kolbe's patent contained cresotic acid. That, however, altogether depended upon the purity or quality of the carbolic acid or phenol from which it was made. The presence of the cresotic acid in the salicylic acid was due to the impurity of the carbolic acid from which it was made. Fischer, in examining a number of commercial samples of salicylic acid made by Kolbe's process found no impurity in several of them.

By Mr. Campbell: Witness had examined other three samples of salicylic acid made by Kolbe's process, and in these there were only faint traces of impurities.

Mr. Ross said that after what had taken place he would withdraw the case.

The Sheriff said that notwithstanding the complaint having been withdrawn it had been a most useful prosecution, as it showed to the profession and the public the results of Professor Charteris's valuable discovery.

Mr. Campbell said that his client and these associated with him desired to the fullest degree to avail themselves of the discovery by Professor Charteris.

THE ANALYST.

DECEMBER, 1890.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A GENERAL meeting of the Society was held at Burlington House on Wednesday, 12th ult., the President, Mr. Adams, in the chair.

The minutes of the June meeting were read and confirmed.

The following gentlemen were declared duly elected as members:—G. A. Milne, F.C.S., Norton; Malton Hugh Barclay, F.C.S., Workington; Edgar Richards, F.C.S., Chemist to the American Inland Revenue.

The following gentlemen were proposed for election:—As members, J. J. Beringer, F.I.C., F.C.S., Analyst for Cornwall; Dr. Readman, Analytical Chemist. As associate, Mr. Gilbard, Assistant to Mr. Dyer, Edinburgh.

The following papers were read:—

"On Food Preservatives."—By OTTO HEHNER.

"On the Adulteration of Food with Boracic Acid."—By CHARLES E. CASSAL.

"On the Detection and Estimation of Boracic Acid in Milk and Cream."—By CHARLES E. CASSAL.

"On the Detection of Methylated Spirits, in Tinctures, Spirits, or other Compounds."—By DR. ASHBY.

The next meeting will be held at Burlington House on Wednesday, 10th December, 1890.

ON THE USE OF FOOD PRESERVATIVES.

BY OTTO HEHNER.

(*Read at Meeting, November,* 1890.)

A FEW weeks ago a milk-seller was summoned before one of the Metropolitan police magistrates for selling milk which was partially deprived of cream, and which, in the words of the summons, contained a poisonous ingredient, namely, boracic acid. The vendor was substantially fined.

This, as far as I am aware, the first case under the Sale of Food Acts in which the use of a food preservative is condemned as an adulteration, opens a question of such importance, that it may well be thought worthy of careful consideration by this Society. We should, as a matter of policy, arrive at some sort of an agreement, and an expression of an opinion, whether we as a body approve of, or condemn, the use of preservatives; and we owe it as public officers to the public and the vendors of articles of food, to let them plainly know what attitude we occupy in reference to a practice which has for years past been allowed without the least objection on our part, and which has become almost universal. It is well known that in summer many milk dealers add some preparation of boric acid in order to retard coagulation of milk. Such preparations are, and have been, openly advertised in numerous forms, and have been recommended by scientific men. Less well known is the fact that boric acid and its soda salt is most frequently to be met with in butter, especially Normandy and Belgian butters. Norwegian and other foreign fish is cured with salt and boric acid; meat is preserved with it, and it is even found in preparations intended for invalids, such as in so-called sterilised peptones.

In beer, especially foreign imported beer, in wines, preserved fruit, and, perhaps, in milk, salicylic acid or salicylate has been used for preservation, whilst sulphurous acid or bisulphite of lime is of common use by the brewer, the manufacturer of lime-juice, and the butcher. And, lastly, it appears that benzoic acid or benzoates are gradually taking the place of salicylic acid, for reasons to which I shall have to refer. Other antiseptics have been from time to time proposed and used, such as fluosilicates, nitrates, etc.

In reference to the quantity of boric acid which is necessary to produce any appreciable effect upon milk, I am not aware of any analytical results obtained by the analysis of actual trade samples. J. Mattern (Ber. der VII. Vers. d. Ver. Bayer. Chem. 36) finds that 1 gram. boric acid per litre retards the coagulation of milk at 15° C. for from twenty-four to thirty-six hours; ·5 gram. only twenty-one hours, whilst at 35° C. ·5 gram. is without effect, and 1 gram. retards the coagulation for ten hours. In summer, therefore, when the addition of boric acid is practised, not much less than 1 gram. per litre of milk would probably be employed.

Of borated butters I have made numerous analyses, and give some of my results in percentages:—

Boric acid	·09	·11	·09	·14	·26	·41
Borax	·19	·16	·15	·23	·33	·55

Both boric acid and borax being calculated as crystallised. The preparation always employed in the case of butter, as far as my experience goes, consists of a mixture of boric acid or borax, in widely varying proportions, freed from a part of the water of crystallisation by heating. As typical the following analysis may find a place:—

Water	36·8
Borax (anhydrous)	24·0
Boric acid (anhydrous)	39·2

To preserve fresh fish about 2 grams. of boric acid are used to each kilo of fish.

Salicylic acid has not, I believe, ever been largely used in this country as a food eserver. It answers but very badly in the case of milk and butter, and its use is now iefly confined to foreign fermented beverages. As to the quantity one gram. per litre as frequently used when salicylic acid had reached its heighth of popularity. When course of time objections against its use were not only heard, but resulted in pro- bitive legislation in many countries, 5 grams. were declared to be sufficient by its vocates to keep 100 litres of Bavarian beer for home consumption, and 20 grams. for port (E. Prior, Deutsche Mediz. Woderschrift, vol. 13, p. 38).

If we now enter into the question as to the physiological effect of the preservatives entioned, we at once meet with the most diametrically expressed opinions. Whilst ventors introduce every antiseptic as absolutely harmless, and quote experiments by hich it is shown that large and continued doses of antiseptic were administered without il effects, other enquirers come to absolutely different conclusions. Literature ems with arguments in favour of and against some of these substances, and it must, I ink, be conceded that direct and palpably injurious effects on healthy individuals have ot been traced to any of the antiseptics herein considered. But when so much is con- ded, a wide field for discussion yet remains.

On the one hand it must be recognised that to prevent waste of good human food by oiding or retarding, its decomposition is a meritorious achievement, and one upon hich the thought of the scientific man cannot be too much concentrated. Yet it must allowed that the indiscriminate addition of chemical substances which exert a isonous action on bacterial and other organisms cannot be safely left in the hands of ore or less ignorant vendors of articles of food, even if no directly poisonous or in- rious action can be traced upon an healthy adult by their use. It is evident that bstances which interfere with the growth of fungoid organisms like bacteria or yeast lls, must have some action upon the complicated human animal; and even if exuberant alth and abundant gastric secretions may be capable many times to overwhelm the fect of the antiseptic, the effect itself must remain and detract from the efficiency of the man organism. It is inconceivable that the protoplasm of a bacteria be vitally fected by a substance which at the same time be utterly inert upon human proto- asm. The effect is evidently a question of quantity. The absolute quantity may not sufficient to show itself palpably upon a body weighing, say, 1½ cwt., but it must be ere all the same.

Well does the Comité Consultatif d'Hygiène Publique de France report in reference the use of benzoic acid in articles of food that all antiseptices are injurious to natural gestion, because the addition of antiseptics of any kind is irrational as far as assimila- n is concerned, and may be injurious to the normal action of the organs of digestion.

In times gone by, when the causes of the decomposition of food were not under- od, the discovery of a preservative was doubtless an achievement; but now, when we t only know these causes, but can prevent decomposition indefinitely by exclusion of rms, or by cold, without the addition of any kind of foreign material, we surely ould make an attempt to discriminate between processes of preservation. If preserva- n could not be effected without the addition of some foreign material, the benefit to mankind of preventing good food substance from decomposition would doubtless be greater than the slight physiological evil effect of the antiseptic itself. But as preservation of any article of food is possible without addition of chemicals, it seems to me that the time has come for public analysts generally to set their faces against the present practice of allowing the addition of any antiseptic which the dealer in food may choose to make.

Isolated action, such as was undertaken by the public analyst to whom I referred in the opening of this paper, appears to me to be most inadvisable, as we would surely have to witness the deplorable spectacle of seeing one analyst giving his opinion against his fellow analyst in a perfectly contrary sense to the destruction of all chance of future legal definition. Of course, it may be that the policy of *laisser-faire* which has hitherto been adopted may commend itself, but if we consider that almost every country in the world has taken this matter up and restricted or prohibited the use of antiseptics in food, whilst England, the country in which food laws were first passed, has done absolutely nothing, it will be admitted that the time is at least ripe for discussion of the subject.

France was the first to raise objections against the use of preservatives. Most of the beer consumed in France used to be imported from Germany. When it was discovered that such beer almost invariably contained salicylic acid, a vigorous agitation, in the interest of French home brewers, commenced. Finally, the Paris Court of Appeal decided that the addition of salicylic acid was to be considered an adulteration, and to the prejudice of the purchaser. The addition was declared to be not harmless, it having been shown that salicylated ales were a source of danger to the community, that salicylic acid was a drug, the use of which had to be ordered by a medical man, and could not be left with the trader. This judgment was based upon a Report of the Commission of the Academie de Medicine of Paris on the Action of Salicylic Acid on Food. The Commmission reported on numerous cases in which the preservation of articles of food by salicylic acid has produced serious results, and is of opinion that it is proved that small but continued doses of salicylic acid or salicylates, may produce serious gastric disturbances, especially with old persons and such who suffer from affections of the liver or organs of digestion. For these reasons the addition of salicylic acid, or salts, should not be allowed to articles of food.

What was originally probably a trade protectionist movement soon became a general opinion. The indiscriminate use of antiseptics should not be allowed, and one State after another passed laws forbidding the addition of preservatives to food, in some cases antiseptics generally, in others specified substances only.

Thus, by order of the Municipal Council of Buenos Ayres of January 5th, 1887, the sale of beer containing salicylic acid was prohibited after March 31st, 1888. Between these two dates the quantity of salicylic acid had to be stated, and was not to exceed ·05 grm. per litre. Other South American States followed. On July 1st, 1887, the town of Milan passed a similar law, referring to the prohibition of salicylic acid in beer, wine, and other articles of food; whilst from July 6th, 1887, the police of Berlin prohibited the addition of any kind of preservative whatever to milk. The town of Schwerin (Mecklenburg) followed suit.

In 1888 the Dutch Government caused an enquiry to be made into the use of salicylic acid in beer, and, as a result, all addition of salicylic acid to food was prohibited. The Italian Ministry, by order of November 29th, 1887, declared the addition of all substances to wine, which were not naturally contained in wine, to be an adulteration; on January 30th, 1888, the Spanish Government followed with an absolute prohibition of antiseptics in wine, and the Austrian with that of salicylic acid.

In Germany, lastly, a distinction is made between salicylic acid added during mashing, to check the growth of the acid-producing organisms, such an addition at that stage of brewing being allowed, not any of the acid, or only traces of it, being said to remain in the beer, and between the subsequent addition of salicylic and fully fermented beer, which is prohibited.

When salicylic acid had thus been virtually suppressed, other antiseptics came into vogue. Thus, in France, the use of benzoates became frequent, partly on account of the greater power of these compounds, partly because they are more difficult to trace and detect than salicylic acid. In Germany sulphites and borates were more largely employed, and numerous preparations containing these as active ingredients were thrown upon the public. Thus, according to Polenske (*Arbeiten des Kais. Gesundheitsamts*, 1870, VI., 119), the following articles have been advertised in Germany :—

Sozolith, containing 39·7 per cent. sulphurous acid, as sodium salt.
Australian Meat Preservative, consisting of sulphites.
Berlinite, borax, with a little boric acid and nitrate of potash.
Chinese Preservative Powder, boric acid, with sodium chloride and sulphate.
Brockmann's Salt, borax, boric acid, sodium chloride, and potassium nitrate.
Australian Salt, borax.
Barmenite, boric acid and salt.
Magdeburg Preservative Salt, borax, boric acid, and salt.
Heydrich's Salt, sodium chloride, potassium nitrate, and boric acid.

Again, as in the case of salicylic acid in previous years, long discussions arose as to the physiological activity of these substances. Liebreich contended that boric acid was perfectly harmless, but others have been able to trace distinct physiological effects to its administration; whilst, at their meeting at Speier, on Sept. 10th, 1889, the Society of Bavarian Analytical Chemists discussed the use of boric acid as an antiseptic, and came to the conclusion that it was objectionable from a sanitary point of view.

In England we have done nothing, absolutely nothing. On the contrary, the use of antiseptics has been virtually sanctioned in brewing. The law 48 and 49 Vict., Cap. 51, declares that a brewer of beer shall not add any matter or thing thereto *except* finings, or other matter or thing sanctioned by the Commissioners of Inland Revenue.

The use of antiseptics, notably bi-sulphite of lime, which is here chiefly used in beer, is therefore distinctly conditional upon the sanction of the Inland Revenue Commissioners; and the same would be the case in other articles of food, since the chemists of the Inland Revenue Commissioners are referees for cases under the Food and Drugs Act. Bi-sulphite of lime being freely used in breweries, with the knowledge of Excise officers, a gentleman wrote to the Board to know if the employment of bi-sulphite of lime and salicylic acid were sanctioned. The reply, which I have seen myself, was that "The

Board do not sanction their use unless they interfere with the Revenue, leaving the responsibility for their use with the brewers."

Surely a remarkable and undignified position to take up. The Act of Parliament says: You shall add nothing to beer, except with the sanction of certain authorities. These authorities say, we leave these matters to the brewers. If the authorities of Buenos Ayres, or of Mecklenburg-Schwerin, were capable of coming to a conclusion, surely it might be reasonably hoped and expected, that the authorities of Somerset House would not remain behind. We public analysts have done no better. The Sale of Food and Drugs Act, par. 6, allows to be added to food any matter or ingredient not injurious to health, if the same is required for the production or preparation thereof as an article of commerce in a state fit for carriage or consumption. No one, surely, can contend that preservatives are necessary for the production or preparation of milk, butter, beer, wine, etc., in a state fit for carriage or consumption, for the great majority of the samples analysed are free from antiseptics. Good milk, fresh butter, sound beer, can be made and sold without antiseptics. As a matter of fact, they have been so sold for centuries, until sham science came in and taught the dirty and the careless producer how to evade the natural punishment of dirt and mismanagement. Antiseptics are convenient to such producers, but they are not *required.* Hence we have no option but to consider them as adulterations.

We might, no doubt, some time longer, as in the past, wink at the use of all preservatives without restriction as to quality or quantity; but we shall, sooner or later, have to answer for our apathy to the public. We might agree between ourselves what limits we would allow, beyond which the quantity of the various antiseptics should not be permitted to rise in food. But the difficulty of exact determination is very great, and, besides, this course would involve the concession of the whole principle, that a dealer in food has no right to add anything whatever to any article of food without due notice, and new antiseptics would be continually introduced, as they have been elsewhere, to circumvent any such partial restrictions. We might further, as another alternative, require that due notice be given by the vendor at the time of the sale that a preservative has been added to the article sold. Here, again, we know from experience how easy it is to circumvent such notice, and how ignorant the public are, and incapable to comprehend the notice. What use, for instance, would it be to inform a servant girl, at the time of sale, that a certain article contains a little boracic acid, or other substance, added from benevolent motives?

Our duty seems plain; we should work for entire prohibition of all kinds of preservatives. It is time that we went back to natural food. I object to be physicked indiscriminately by persons not qualified to administer medicine whilst I am in health; I object still more when I am ill. I object still more strongly to have my children physicked in their milk, or their bread and butter. It is no consolation to me to know that the physic is not immediately fatal, or not even violently injurious.

The practice is utterly unjustifiable except from the point of view of a dealer, who wants to make an extra profit, who wants to palm off a stale or ill-prepared article upon the public.

We owe our very existence, as public analysts, to the desire of the public to have a guarantee of the absolute purity of their food supply, and we would only be doing our duty if we did suppress the large and growing use of these objectionable chemicals called food preservatives.

ON THE ADULTERATION OF FOOD WITH BORACIC ACID.

By Charles E. Cassal.

Read at Meeting, November, 1890.

The increasing use of boracic acid, as well as of other so-called antiseptic agents for the preservation of articles of food, is a matter which demands immediate and most serious attention from public analysts. Inasmuch as my friend Mr. Hehner, with whom I have had several conversations on the subject, has consented to give the Society the benefit of his wide experience in this matter, I have thought that it would be convenient for the remarks which I should have to make upon it to be placed before the members in the form of a short paper at the same time. I propose, on this occasion at any rate, to deal with boracic acid only, because I have given some amount of attention to its use, and because I believe I was the first public analyst to officially certify milk and cream which contained it, as adulterated within the meaning of the Act. My first certificate was issued in 1887 upon a sample of cream which contained boracic acid; the local authority did not deem it advisable to take action at that time, and referred the matter to the Medical Officer of Health and to me, with the request that we should draw up a joint report to advise them whether the addition of boracic acid to milk and cream was injurious to health. This report was duly drawn up and presented. The matter excited some attention at the time, but, for reasons which I need not enter into here, no prosecution for adulteration with boracic acid was then undertaken.

Boracic acid is generally added to milk and cream in the form of certain proprietary articles, which are sold to the trade under different fancy names. Some of the more commonly used preparations, which consist of a mixture of boric acid (partly anhydrous and partly hydrated) with borate of soda, are added (in solution) in the proportion of *at least* 7 grains of the solid substance to the pint of milk. In cream it is generally added in *at least* double this quantity per pint. Inasmuch as both the farmer in the country and the dairyman in London are likely to use these preparations, these amounts may be largely increased. The amounts mentioned are also very likely to be exceeded through the carelessness or ignorance of those making use of such substances for preserving purposes.

But scanty information is to be obtained as to the action of boracic acid and borates on the human subject.

These substances do not appear to have been much used as internal remedies, and, accordingly, but little information as to their action is to be found in the text-books. One authority states that borax acts as a mild alkali on the alimentary canal; tending to render the fluids alkaline, and to cause diuresis; that it checks fermentation due to organisms, and is used as a diuretic and antacid. Another eminent authority who was consulted in connection with the report previously alluded to, refers to the tendency of these substances to set up diarrhœa, a disease very prevalent in hot weather, when the preservatives are most used to prevent the change which milk and cream are apt to undergo rapidly at a high atmospheric temperature. The medicinal dose of the acid as laid down in the British Pharmacopœia for an adult ranges from 5 to 30 grains, and, on the authority of my colleague, Dr. Dudfield, and of others, it may be computed that one-twelfth of the quantity, say a maximum of 2½ grains would be a suitable dose for a child under one year. It is quite possible that a strong, healthy infant, taking a quart of milk daily might absorb as much as 20 or 25 grains in that time, having regard to the likelihood of the minimum amounts mentioned being exceeded.

The experiments of Dr. J. Förster, published some years since in a German scientific journal,* go to show that the addition of boracic acid to articles of food in far smaller proportions than is customary is injurious to health. This author considers that "even small doses of it are injurious to the digestive organs." The injurious action, he says, depends on the circumstance that the drug acts so as to materially increase the proportion of solid matters and nitrogen in the fæces separated. Its action on the intestinal discharge is well marked, even by the exhibition of as little as 4 grains per diem, and is stated to be in direct relation to the quantity taken, and to be maintained for some time after the doses of the drug have ceased. The action described is perceptible, not only with vegetable or animal foods, which contain a large proportion of indigestible ingredients, but also when highly-digestible foods, such as milk and eggs, are taken. Food to which the acid has been added tends to cause an increase in the secretion of gall during assimilation. One of its most important actions, however, according to this author, is the increase which it causes in the discharge of albuminous substances from the intestinal canal.

E. Hötter has recently published a series of experiments on the action of boron compounds on plants. The absorption of such compounds was found by him to destroy the chlorophyll, and hence to arrest the processes of assimilation. The roots are affected, and soon die. Free boric acid was found to be more prejudicial than the alkali salts.

Apart from the experiments referred to, it is sufficiently plain that an "antiseptic" substance of any kind, if introduced into food must, by its very nature, be injurious to the health of the consumer. It is evident that the introduction of antiseptics of any kind into the alimentary canal must upset the conditions necessary to health. The danger is the greater because it is insidious, and because the effects produced are not violent or sudden, but such as will create disturbances and ultimately serious injury, which are likely to be ascribed to anything but the real cause. I consider that the question of quantity does not bear upon the matter in any way. I hope to show that it has, or should have, no bearing legally. Any quantity that is sufficient to produce the "antiseptic" effect in the food itself, must be regarded as injurious, even if the undoubtedly harmful action of continued small doses of a "preservative" substance be left out of consideration. It must be remembered that boracic acid preservatives are very extensively used. In butter, milk, cream, fish, meat, and in preserved foods and proprietary preparations of various kinds, boracic acid is to be constantly found. I have recently found it to be present in large amounts in meat "extracts" and "fluids" intended especially for the use of invalids and children, and which, I regret to say, are belauded in inflated language by members of the medical profession, who, *had they known* of the presence of boracic acid, would surely have condemned them. The unavowed presence of boracic acid in such preparations, or in the milk and cream so largely used by invalids and children might, and, as pointed out by a writer in the *British Medical Journal*, in most cases probably would seriously disturb a physician's course of treatment.

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* Dingler.

Attempts have been made to confine the issue by asserting that it is as permissible to add boracic acid to a food as it is to add salt in certain instances (as in the case of salt meat). This contention is at once disposed of by pointing out the physiological fact that salt is a *food*, and that boracic acid is not. Again, boracic acid, and compounds of it, are not natural constituents of milk or of any food. They are not themselves "foods," but "drugs."

Proceedings could be taken against the vendors of milk containing boracic acid compounds under the 3rd Section of the Sale of Food and Drugs Act, which prohibits, with certain unfortunate reservations, the "mixing, colouring, staining, or powdering" of any article of food with any ingredient or material *so as to render* the article injurious to health. It is a matter of great difficulty, however, to prove "injury to health" to the satisfaction of an ordinary court. It is almost necessary in all such cases to be able to produce some poor creature who has been almost "done to death" by that which is complained of. Under this section also it is practically a necessity to state the percentage of the injurious substance; to afford, of course, a useful bone of contention for members of the legal profession, and not to obstruct in any way the wide loop-hole for the escape of offenders, duly left by the legislature, in this section. I contend that it is unnecessary to state percentage in a matter of this kind. The 6th Section of the Act makes it an offence to sell to the prejudice of the purchaser any article of food which is not of the "nature, substance and quality" of the article demanded by the purchaser, and provides that no offence is committed under this section if "any matter or ingredient not injurious to health" has been added, because such addition is required for the production or preparation of the food as "an article of commerce," or "in a state fit for carriage or consumption," and "not to conceal the inferior quality thereof." The 2nd Section of the Amendment Act of 1879 provides that it is no defence to prove that the "article of food in question, though defective in 'nature,' or in 'substance,' or in 'quality,' was not defective 'in all three respects.'" That is, proceedings would be justified by a defect in any one respect.

There appears, however, to be an impression abroad that there is no offence under the 6th section unless the adulterant forms "a substantial portion"—that is, presumably, more than a fractional part of the article as sold to the purchaser. Taking the case of milk, it ought hardly to be necessary to point out: (1) That milk containing boracic acid compounds is not of the nature, substance and quality of *milk*; (2) That even if defect in *nature* and *substance* is only constituted by the presence of an adulterant in what is popularly called "a substantial amount" (that is, forming a large integral portion of the article), defect in *quality* is at once established by the presence of boracic acid in milk, and that such defect is independant of the question of quantity; further (3) the admixture of boracic acid compounds with milk is not *required* for preserving it in course of transit from producer to consumer. Refrigeration, which adds nothing to the milk, is the recognised and legitimate method, if such preservation for transit be deemed necessary; and (4) the addition of the preservatives does serve the purpose of concealing the *inferior quality* of the milk, as it allows stale milk to be supplied as fresh, and as it is far more easy and less expensive than the better method of refrigeration.

I therefore hold that vendors of foods containing boracic acid compounds commit offences under the 3rd and the 6th Sections of the Act of 1875, and the 2nd Section of the Amendment Act of 1879. Whether offences under the Act, however, are committed or not, I do not see how it is possible to arrive at any other conclusion but that the use of these preservatives is a serious form of adulteration. In my view, the absolute prohibition of their use is the only safe and proper course to take; although if it be desired to throw a sop to that large class of persons who are always morbidly anxious to follow middle courses, it may be admitted that the compulsory labelling of such "preserved" articles with the name of the substance or substances used, would be productive of great and immediate benefit. It is remarkable that those who defend the use of "preservatives," and assert them all to be "harmless," in any amount, should take such pains to conceal the fact that they have been used. In regard to several proprietary articles I have examined; I have had before me, apparently, the full details of the manufacture, given with the most child-like candour; everything, *except* the admixture of boracic acid, which was found in them all. In the case of certain "preserved" creams, it is asserted on the labels that "the cream being separated as soon as it is drawn from the cow, it will keep sweet longer." Comment upon this is needless.

Abroad, the use of "preservatives" is almost universally prohibited. If the legislature and if local authorities were in earnest about the suppression of adulteration, their use would be prohibited here. In any case, it is our duty as public analysts to fully advise those who appoint us about what is being done. The *onus* for not taking action will then not rest with us.

DETECTION AND ESTIMATION OF BORACIC ACID IN MILK AND CREAM.

By Charles E. Cassal.

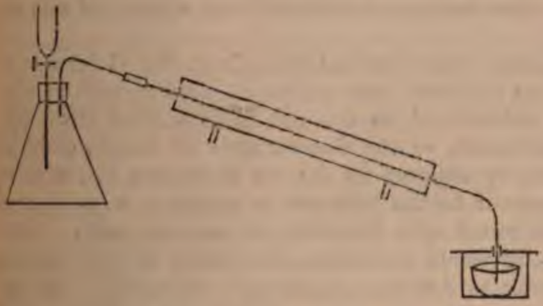
(*Read at Meeting, November,* 1890.)

The presence of boracic acid and borates in milk and cream can be detected without difficulty by applying the turmeric test and the alcohol-flame test to the ash.

The ash obtained in the course of the ordinary analysis is treated with a little water and made just acid with hydrochloric acid. Numbered slips of turmeric paper are moistened half way with the solution, and are preferably arranged on a glass plate and dried in the air bath; or a few drops of turmeric solution are added to the acidified ash, and evaporated to dryness in a porcelain capsule, the latter method being an exceedingly delicate one. On moistening with an alkali, the dark blue colour is obtained on the paper or porcelain when boracic acid is present. A dark blue solution is obtained when water and an alkali are added to the residue in the porcelain capsule, but the colour disappears with great rapidity, and I have hitherto been unsuccessful in determining the conditions which would render it permanent, or sufficiently permanent to admit of its being applied for purposes of estimation. The only method which up to the present I have found to be applicable for the estimation of boracic acid in milk and cream is a modification of that described by F. A. Gooch, in the *American Chemical Journal*, and which depends on the fact that free boracic acid can be completely separated from fixed substances by repeated distillation with methyl alcohol. I have repeated Mr. Gooch's experiments, and am able to confirm his results generally. He states that methyl alcohol, ethyl alcohol and water are effective for the purpose of separation in the order named, and this I have found to be the case; but the volatilisation of even 0·2 grams. of anhydrous boric acid by the use of ethyl alcohol is so tedious that it is not advisable to use this substance for the purpose. Complete volatilisation of boric acid by the use of successive charges of methyl alcohol is easily and rapidly effected. I have found it best to use successive charges of 5 c.c. rather than 10 c.c., or more, as used by Mr. Gooch. I am not able to confirm his statement that in the presence of water methyl alcohol is not equally effective. My own experiments show that crystallised boracic acid is volatilised more rapidly than anhydrous boric acid, and that the addition of a few drops of water to the latter before evaporation or distillation with methyl alcohol promotes rather than retards the volatilisation of the acid.

The method as now applied by me for the estimation of the acid in milk and cream is as follows:—In the case of cream about 50 grams., and in that of milk about 100 grams. of the sample are rendered alkaline with caustic soda, evaporated to dryness and incinerated. The ash, which need not, of course, be burnt white, is ground up and transferred, washing in with a little methyl alcohol and a few drops of water, to a conical flask of from 200 c.c. to 300 c.c. capacity, provided with a doubly-perforated caoutchouc cork, through which pass a stop-cock funnel tube and a delivery tube. The flask is attached to an ordinary condenser by means of a flexible joint in such a way as to admit of its being shaken round occasionally, and is placed in an oil-bath. The mixture in the flask is acidified with acetic acid, and 5 c.c. of methyl alcohol is run in from the funnel tube, and distilled. The distillate is received in a weighed amount of pure lime (ignited to constant weight). About 1 gram. is a convenient quantity. The lime is contained in a platinum dish capable of holding about 70 c.c., which is placed in a glass receiver.

This receiver may be made from a ground-edged glass vessel, covered with a perforated ground-glass plate. The end of the condenser tube passes through a grooved or loosely-fitting cork in the perforation of the plate, and terminates just above the lime in the platinum dish.



Distiliation with 5 c.c. of methyl alcohol is repeated several times, ten such treatments being ample for all likely cases. Fewer are generally quite sufficient. A gelatinous mixture is obtained in the platinum dish. It is well stirred, and allowed to stand for some minutes. It is then evaporated in an oil bath, and is finally heated over the blow-pipe flame to constant weight. The increase of weight is boric acid, and the error does not in any case amount to more than 1 milligramme *plus* when dealing with from 0·1 to 0·25, or 0·3 grams. of anhydrous boric acid. To make sure that the acid has been completely volatilised, the residues in the distillation flasks are tested with turmeric paper.

The few drops of water added before the first distillation greatly increases the rapidity of the removal of the boric acid. The use of nitric acid is not advisable for the purpose of freeing the boric acid in the flask. Acetic acid presents no difficulties, and is quite effective.

There is no advantage to be gained, in my opinion, from the use of the special apparatus described by Mr. Gooch in his paper, and the difficulties and inaccuracies attendant upon transference of the lime from crucible to flask, and from flask to crucible, are got over by carrying out the whole operation in one platinum dish.

My thanks are due to my assistant, Mr. Gerrans, for the care and trouble he has taken in helping me to carry out the necessary experiments.

DISCUSSION.

MR. DAVIES said it appeared to him that the two papers they had just listened to opened up a large field for the consideration of public analysts, and personally he felt very much indebted to the two gentlemen who had brought them forward, particularly for the information that had been given with regard to the physiological effect of the boracic acid and borates, and for the general resumé of the whole field of information upon the subject. The fact was new to him that so many other states and countries had legislated on that particular subject, and that fact in itself was a very important reason in favour of there being legislation upon the matter here. He, personally, was disposed entirely to agree with the authors in the conclusions they arrived at that such additions of preservative agents are to be regarded as adulteration. That was the view he himself had held. He had asserted the presence of boracic acid in one or two cases, and although he had never actually certified to his vestry that it was an adulteration that called for their action, yet at the same time, when he had been asked in his capacity of analyst by private individuals, who had sent him samples, if he would regard such addition as adulteration, he had distinctly stated that such was his opinion, and that if such samples came before him in his capacity of public analyst they might expect that he would certify them to be adulterated in consequence of that addition. The papers would be exceedingly useful if they served to elicit the opinions of such public analysts as were present with regard to the question.

MR. BLOUNT said that salicylic acid had been shown to be of different properties, according as it was natural or artificial, and possibly the injurious physiological action that had been noted was due to the fact that experiments had been conducted with the impure artificial variety.

MR. ALLEN said that he fully agreed with what had fallen from Mr. Hehner as to its being the duty of public analysts to insist that articles of food should be what they represented to be. On the other hand he was not fully satisfied that some of these preservatives were so objectionable as the Governments of certain countries would seem to think; but he distinctly objected to the use of boracic in milk, as its addition was a direct encouragement to the milkmen to separate a portion of the cream and sell the partially skimmed milk the next day as new milk. Hence the detection of boracic acid in milk would strengthen the hands of the analysts who had occasion to report on a milk as deficient in cream or butter-fat. In fact, it was a fair inference from the presence of boracic acid that the milk was not new milk. With regard to the use of salicylic acid as a preservative, the difference in physiological effects of the natural and artificial acid might be safely ignored as in the small amounts of salicylic acid used it would be immaterial whether the salicylic acid was perfectly pure, or whether it contained a small admixture of cresotic acid such as often occurred in the commercial artificial acid. The use of salicylic acid for preserving beer seemed a less serious matter than its addition to milk. Fortunately salicylic acid could be detected and separated with perfect certainty from matters containing it, and the same was true of benzoic acid. As to boracic acid, he congratulated Mr. Cassal on bringing before them so ready a method of estimating it. The method, though not strictly original, was little known to the majority of analysts.

Mr. Allen said he sympathised with any analyst who acted the part of pioneer. With regard to what had been said as to consultation with brother analysts before taking any decided action, he might remind them that the Society had not met for a period of five months, which was an undesirably long time. He thought the paper and comments on it showed the great value the Society had for its members.

Mr. Adams said that no better illustration could be given of the value of their meetings than that afforded by the papers just read by Messrs. Hehner and Cassal, and the remarks of the members present that had thereby been elicited; in all cases where a question arose such as these papers brought forward, it was a good thing that the views of the profession as a body could be ascertained, in order that some unanimity of action might follow, and mutual support and confidence fostered when circumstances necessitated new lines of action in reference to the official duties of the public analyst.

His own opinion with reference to the point at issue was that Mr. Cassal had used rather too strong a term when he said "*poisonous*"; but although boracic acid may not be poisonous in a general sense it did not follow that it may not sometimes be injurious —its effect probably might vary, and depend very much upon the individuals operated upon. It was well known that some drugs which act favourably on most people, seriously disagree with others, and even in ordinary matters of food it was astonishing what a variety of opinions were held by various people regarding the wholesomeness and digestibility of various food substances. He himself knew of a remarkable case of a gentleman who always suffered in a surprising manner after eating mutton—this food which is commonly regarded as being particularly easy of digestion, acted upon him as a violent poison—causing him to vomit, and to be purged; at first it was set down to some mistake or fancy on his part, but no, let the mutton be disguised in any way by mixture with other things, or by the addition of condiments, the same result always followed its ingestion, so that one might contend that it was impossible to predicate with certainty that any particular food or drug would be equally acceptable to all persons. In the speaker's opinion, therefore, in every case of preserved food it should be stated that such and such a food was preserved by the addition of salicylic acid or what not, so that the consumer might know what he was about to use. Moreover, he did not think any substance should be admitted into general use until its innocence had been established, until then it should be looked upon as an injurious, if not poisonous substance, and he thought this seemed to be the general opinion of the members of the society present.

Dr. Veith said that he was very strongly of opinion that, so far as the London trade was concerned, there was no reason why fresh milk and cream should contain boracic acid, the best proof being that large quantities were sold daily to which no preservative had been added. But if someone went to any grocer's, poulterer's, cheesemonger's, or other shop, asking for potted cream which was expected to keep sweet for several days, and if that cream was found to contain boracic acid, he—Dr. Veith—must maintain that such cream certainly was of the nature, substance, and quality of the article demanded.

Mr. Cassal said that the case which had been alluded to by Mr. Hehner was one in which he had certified a milk as having had at least 30 per cent. of the original fat abstracted, and as containing boracic acid. The authority had, therefore, not prosecuted on that point alone, but it was a part of the complaint as stated in their summons. The case was heard at petty sessions, and he was informed that the Chairman of the bench of magistrates in convicting the defendant severely reprimanded him, and made some strong remarks about the boracic acid adulteration. He certainly considered it most desirable that the public analysts of the country should fully discuss all matters of this kind, and that they should be as far as possible agreed. Squabbles between professional men in a law court were most injurious and discreditable. Public analysts did not act sufficiently in concert, and this was still one of the great evils of the profession, although matters were now much better than they used to be in this respect. But it was not always possible to consult one's colleagues, or to get a particular subject adequately discussed and a definite decision arrived at. It was plainly the public analyst's duty to certify milk containing boracic acid as adulterated. He could not act otherwise. The public authority who received the certificate might or might not take action upon it. This was no part of the analyst's business. If they took action he could not stand in the way of their doing so. He hoped that the Society would arrive at some decision in the matter.

Mr. Hehner said that he had not the remotest intention to express any censure upon Mr. Cassal for having taken action against a vendor of preserved food; on the contrary, he was thoroughly in accord with Mr. Cassal's opinion. But after public analysts had allowed, and almost encouraged, the use of food preservatives for many years—their open sale was no secret—it was rather hard now to suddenly turn round and to say to the dealers, "you have added a poisonous or injurious ingredient to the food you sold." As in the case which he mentioned in the beginning of his paper, there was abstraction of cream, the magistrate naturally did hold that this was aggravated by the addition of preservatives. He thought it would be desirable to select, as a test case, one in which no ordinary adulteration had been practised, such as the addition of boric acid to cream, or to otherwise genuine milk.

In his opinion, the injuriousness and otherwise of antiseptics was altogether beside the question. Granted that in at least 99 per cent. of food preservatives did no harm, the addition being unnecessary, was illegal under the Sale of Food Act. It should not be forgotten that in the process of digestion, bacterial organisms, especially in the lower portions of the intestines, played an important part in helping to break up such food matters as had escaped solution by gastric and pancreatic ferments. Food preservatives were added with the avowed object to prevent the growth of bacterial organisms, but we had a perfect right to object against the destruction of our intestinal bacteria, which, in all probability, were necessary aids to our nutrition, by our milkmen, buttermen, butchers and brewers.

Dr. Adams had alluded to the fact that certain persons might be affected by some preservatives, whilst others were not. In our ordinary feeding we all know this to be the case. If a person, for instance, from his experience knew that pork disagreed with him, he naturally would avoid eating it. But if a person were more or less affected by boric acid or other preservatives, how could he avoid it, as it was presented to him in so many articles of food without any notice whatever?

(*Conclusion of the Society's Proceedings.*)

A METHOD FOR THE ESTIMATION OF ALBUMEN IN URINE.

By T. C. Van Nuys and R. E. Lyons.[*]

That albuminous bodies are completely precipitated from solution by tannic acid is proven by the investigations of Liborius,[†] Girgensohn[‡], Sebelien[§], and others. By

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[*] "American Chemical Journal."

[†] Beitrag zu quant. Eiweissbestimmungen. Dorpat, 1871.

[‡] Beiträge zur Albuminometrie. Dorpat, 1872.

[§] Studien über die analyt. Bestimmungsweise der Eiweisskörper mit besonderer Rücksicht auf die Milch.

Sebelien's method of estimating the total quantity of albuminous bodies in milk, 3 to 5 grams. milk is diluted with some water, a few drops of a solution of sodium chloride or magnesium sulphate added, and the fluid is then treated with an excess of Almén's solution of tannic acid,* the precipitate filtered off, well washed, and the nitrogen in the precipitate estimated by Kjeldahl's method.

The weight of nitrogen is multiplied by 6·37, the product of which is the weight of albuminous bodies in the weight of the milk taken. The factor 6·37 is the number of times the per cent. (15·7) of the nitrogen in albumen is contained in 100 $\left(\frac{100}{15\cdot7}=6\cdot37\right)$.

Sebelien's method differs from the methods of Almén and Liborius in not depending on the fixed constitution of the compound of albumen and tannic acid, but rather all of the albumen is separated from solution, and that all nitrogen compounds not albuminous can be removed from the precipitate by washing.

Our studies of this subject were carried on with the view of ascertaining if a method of estimating albuminous bodies in urine, employing tannic acid, is practical. As urine contains nitrogenous compounds in considerable quantities, the tannic acid compound of albumen is not easily washed until pure; besides, by continued contact of an acid, or acid salts, uric acid is precipitated, which would remain in part with the albumen; and as the volumetric method of estimating albumen with tannic acid has not been found accurate†, if there remains a practical method for estimating albumen in urine with tannic acid, it is that the total quantity of nitrogen in albuminous urine is first estimated, following which estimation is made of the quantity of nitrogen in the urine having its albumen separated by means of tannic acid. The difference in the quantities of nitrogen found is the weight of nitrogen of the albumen, which multiplied by 6·37, would give the weight of albumen. To determine if the method here outlined yields accurate results, the question arises if uric acid or other nitrogenous compounds of normal urine are retained by the precipitate formed by the tannic acid and albumen when filtered, so that the volume of urine in the undiluted filtrate would contain a smaller quantity of the normal nitrogenous constituents of the urine than is in a corresponding volume of urine before the separation of albumen.

To determine this we estimated the nitrogen in normal urine before and after the introduction and removal of known quantities of albumen. We determined if Almén's solution of tannic acid produces a separation of any part of the nitrogenous compounds of normal urine, or of urine containing an abnormal quantity of the urates, and finally we employed the new method and the gravimetric method in estimating the albumen in urine and compared the results.

Pure albumen was prepared by dissolving the whites of eggs in a small quantity of water, saturating the solution with magnesium sulphate and filtering. The filtrate was then treated with dilute acetic acid until a precipitate ceased to form, filtered off, and the precipitate washed with a saturated solution of magnesium sulphate.

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* 4 grams. tannic acid, 8 c.c. acetic acid (1 part glacial acetic acid and 3 parts water), 190 c.c. dilute alcohol, 50 per cent.

† Beiträge zur Albuminometrie von Girgensohn; Dorpat, 1872. Einige Methode zur Werthsbestimmungen der Milch von Tarazewiscz; Dorpat, 1873.

Having pressed the precipitate between porous paper, it was dissolved in a small quantity of water, the solution neutralised with sodium hydrate, and dialysed until nearly all of the magnesium sulphate was separated. To the turbid solution of albumen a small quantity of the oil of thyme was added to prevent bacterial decomposition, and having mixed well, the solution was filtered. In case a solution was too dilute it was concentrated by evaporation at 40° to 50° C., and when nearly free of ash, some sodium chloride was added to facilitate the precipitation with tannic acid. Solutions of ovalbumen we number 1, 2, 3, etc.

FIRST ALBUMEN SOLUTION.

Estimation of Albumen.

(1). 10 c.c. albumen solution, 10 c.c. water mixed.

(*a*). 5 c.c. of the solution required by Kjedahl's method 47·3 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the solution required by Kjeldahl's method 47·3 c.c. $\frac{1}{5}$ normal KOH, corresponding to 0·1512 gram. nitrogen in 100 c.c of the undiluted solution of albumen.

(2). 10 c.c. albumen solution, 10 c.c. Almén's solution of tannic acid mixed and filtered without dilution.

(*a*). 5 c.c. of the filtrate required by Kjeldahl's method 49·9 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the filtrate required by Kjeldahl's method 49 95 c.c. $\frac{1}{5}$ normal KOH, average 49·925 c.c., corresponding to 0·0084 gram. nitrogen of bodies not albuminous in 100 c.c. of the albumen solution, The quantity of nitrogen of bodies not albuminous deducted from the total quantity of nitrogen, 0·1512 gram. leaves 0·1428 gram. nitrogen of albumen, corresponding to 0·90963 gram. albumen in 100 c.c.

Urine.

(1). 10 c.c. normal filtered urine, 10 c.c. water mixed.

(*a*). 5 c.c. of the diluted urine required by Kjeldahl's method 3·58 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the diluted urine required by Kjeldahl's method 35·8 c.c. $\frac{1}{5}$ normal KOH, corresponding to 1·5904 gram. nitrogen in 100 c.c. of the undiluted urine.

(2). 20 c.c. urine, 10 c.c. albumen solution, 10 c.c. Almén's solution of tannic acid, mixed well in a small flask, and filtered without diluting.

(*a*). 5 c.c. of the filtrate required by Kjedahl's method 35·75 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the filtrate required by Kjeldahl's method 35·8 c.c. $\frac{1}{5}$ normal KOH, average 35·77 c.c., corresponding to 1·5932 gram. nitrogen in 100 c.c. of the undiluted urine; but as there was 0·0084 gram. nitrogen in 100 c.c. of the albumen solution which was not of the albumen, and therefore not precipitated by tannic acid, and the volume of No. 2, 5 c.c. is $\frac{1}{4}$ albumen solution, there is $\frac{1}{4}$ of 0·0084, 0·0021 gram. nitrogen bodies not albuminous of the urine, and consequently 1·5932 — 0 0021 = 1·5911 gram. nitrogen in 100 c.c. urine. Therefore there was a gain of 0·0007 gram. nitrogen by the process, 1·5911 — 1·5904 = 0·0007.

SECOND ALBUMEN SOLUTION.

Estimation of Albumen.

(1). 10 c.c. albumen solution, 10 c.c. water.

(*a*). 5 c.c. of the diluted solution required 47·9 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the diluted solution required 48·0 c.c. $\frac{1}{5}$ normal KOH, average 47·95 c.c., corresponding to 0·2296 gram. nitrogen in 100 c.c. of the undiluted albumen solution.

(2). 10 c.c. albumen solution, 5 c.c. Almén's solution, 5 c.c. water, mixed well in a small flask and filtered.

(*a*). 5 c.c. of the undiluted filtrate required 49·95 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the undiluted filtrate required 49·95 c.c. $\frac{1}{5}$ normal KOH, corresponding to 0·0056 gram. nitrogen of bodies not albuminous in 100 c.c. of the undiluted albumen solution. Deducting the latter weight from the weight of the total quantity of nitrogen (0·2296 — 0·0056 = 0·224), there is 0·224 gram. nitrogen of the albumen in 100 c.c. of the solution, which corresponds to 1·42688 gram. albumen in 100 c.c. (0·224 × 6·37 = 1·42688).

Urine I.

(1) 10 c.c. normal filtered urine, 10 c.c. water.

(*a*). 5 c.c. of the diluted urine required 37·3 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the diluted urine required 37·35 c.c. $\frac{1}{5}$ normal KOH, average 37·325 c.c., corresponding to 1·4196 gram. nitrogen in 100 c.c. of the undiluted urine.

(2). 10 c.c. urine, 5 c.c. Almén's solution, 5 c.c. water, mixed well in a small flask and filtered.

(*a*). 5 c.c. of the filtrate required 37·25 c.c. $\frac{1}{5}$ normal KOH.

(*b*). 5 c.c. of the filtrate required 37·3 c.c. $\frac{1}{5}$ normal KOH, average 37·275 c.c., corresponding to 1·4252 gram. nitrogen in 100 c.c. undiluted urine.

Deducting $\frac{1}{4}$ of 0·0056 gram., the weight of nitrogen of bodies not albuminous, there remains 1·4238 gram. nitrogen in 100 c.c. of the undiluted urine. The results show a gain of 0·0042 gram. nitrogen in 100 c.c. of the undiluted urine after the separation of the albumen, 1·4238 — 1·4196 = 0·0042.

(*To be continued.*)

REPORT OF RECENT RESEARCHES AND IMPROVEMENTS IN ANALYTICAL PROCESSES.

ON COCO-NUTS. L. VAN ITALLIE. (*Ned. Tydschr. v. Pharmacie*). October, 1890.—A large nut contained about 155 grammes. of milky fluid of 1,022 specific gravity (at 18° C.) The reaction was faintly acid, and no precipitate was obtained on boiling or on addition of acetic or picric acids, showing the practical absence of albumenoids, which might have been expected in a so-called milk. 100 c.c. of the fluid were evaporated to a syrupy consistence and extracted with alcohol. The precipitate was washed on the filter with alcohol, but it readily dissolved in a little water. Delicate tests showed no traces of albumen, but the liquid readily reduced Fehling's solution. The filtrate was evaporated to a very small bulk, once more treated with alcohol, and the precipitate finally dissolved in water. The solution was not precipitated by picric acid, Mayer's solution, phospho-tungstic acid, potassium-cadmium iodide, platinic chloride, auric chloride, and tannic acid. Neither lead acetate nor the basic salt gave any precipitate, and no alkaloidal or glucosidal body was extracted by agitation with ether, chloroform, or benzol. 280 grammes of the endocarp were grated and pressed out. The fluid was precipitated with lead acetate, and the filtrate treated with liquor plumbi. The precipitate was washed, suspended in alcohol, and decomposed with hydrogen sulphide. The filtrate from the lead sulphide was nearly colourless, and gave after evaporation no precipitate with the various general reagents for alkaloids. After boiling with dilute sulphuric acid, it did not reduce Fehling's solution, showing the absence of glucosides. The filtrate was freed from lead by a current of hydrogen sulphide, and evaporated to a syrupy consistence after neutralising the liberated acid. The liquid was mixed with alcohol, filtered, the alcohol evaporated, and the residue dissolved in water. The various reagents for alkaloids failed to produce any precipitate. Fehling's solution was reduced, but after removing the suboxide of copper, and boiling with dilute sulphuric acid, no further power of reducing copper solution was noticed, showing to the author's satisfaction the complete absence of any *alkaloids* or *glucosides* in the coco-nuts. To ascertain the nature of the sugars in the nuts, the author mixed 50 grammes. of the juice with a very slight excess of ammoniacal * liquor plumbi. The filtrate showed undoubtedly the presence of sucrose by the usual tests. The lead precipitate was after washing suspended in water and treated with carbonic anhydride. The filtrate strongly reduced the Fehling, showing the presence of dextrose. The residue, which now might have contained the lead compound of levulose, was decomposed by hydrogen sulphide; but as the filtrate, after evaporation and neutralisation, did not reduce Fehling's solution, the absence of that substance may be taken for granted. L. DE K.

ADULTERATION OF STARCH. F. DICKMANN. (*Zeitschr. f. Angew. Chemie.*) 1 Oct., 1890.—The author, in testing a sample of starch for ash by incinerating it in a platinum crucible, found his dish completely perforated, and to contain a lump of a metal. This consisted of lead, which was present in the sample as sulphate, to the extent of 17·6 per cent. Other mineral matter could not be detected. The moisture of the sample amounted to 11·75 per cent., and under the microscope the starch seemed to be all rice. The sample was not in powder but in the usual lumps, so there could be no question of an accidental contamination. The author is at a loss to understand what might have been the object of introducing a rather expensive article, such as lead sulphate, which has not even the power of improving the colour of the article. L. DE K.

DETECTION OF MARGARINE IN BUTTER. BOCHAIRY. (*Ph. Zeit.*, 518).—Fifteen c.c. of the filtered butter-fat are put into a graduated cylinder, dissolved in 15 c.c. of toluene and 50 c.c. of commercial absolute alcohol are added. If the temperature does not exceed 18° C., the spirit will float on the top of the solution of the butter. The liquid must now be heated up to 50° C., and then thoroughly shaken. If the sample consists chiefly of margarine, the liquid gets at once turbid; but when one has to deal with a mixture, there forms no sensible deposit until the cylinder is put into water of 40° C. temperature. If the butter is adulterated, a more or less heavy layer will separate out. For instance, with a sample containing only 10 per cent. of margarine, 11 c.c. of deposit were obtained, but, unfortunately, the amount does not increase proportionately with the amount of margarine present. However, as a qualitative test, the process certainly deserves a fair trial. L. DE K.

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* *Liquor* plumbi mixed with ammonia until a permanent precipitate threatens to form.

Assay of Commercial Caustic Soda. G. Lunge. (*Zeitschr f. Angew Chemie*, tober, 1890).—As is well known, commercial caustic soda contains some soda com- unds capable of saturating acids, which are consequently reckoned as sodium oxide. ese compounds are sodium carbonate, silicate, or aluminate. In accurate commercial alysis the amount of sodium carbonate is generally calculated from the amount of rbonic anhydride the sample contains; but there seems to be a kind of unwritten reement not to pay attention to the fact that some of the caustic soda found really ists as a silicate or an aluminate. This is most likely, because in practice (soap aking, etc.), the presence of these compounds does not interfere with the manufactur- g; and they are also generally present in but trifling quantities. The author's process titrating with methyl-orange has been criticised by Cross and Bevan, who hold that e process is wrong because some of the alumina will also count as soda. The author ints out that these chemists made a mistake in supposing two molecules of alumina to utralise five molecules of sulphuric acid instead of six; and also that their idea about e interference of alumina is much exaggerated. The author has rarely found the nount of alumina to exceed ·1 per cent., which is not astonishing, as the high mperature to which caustic soda is exposed during the fusion makes it deposit nearly l its impurities. Of course, the so-called caustic bottoms may contain sometimes as uch as two per cent. of alumina, and cannot very well be titrated with the aid of ethyl-orange.

L. de K.

Quantitative Estimation of Fluorine. H. Offermann. (*Zeitschr f. Angew hemie*, No. 20, 1890).—The author's process does not much differ from the one scribed in The Analyst, 1886, by Dr. S. Bein; but instead of weighing the separated licic acid, the fluid is titrated with normal alkali, with tincture of cochineal as indi- tor. As 6 molecules of potassic hydrate = 1 molecule of hydrofluosilicic acid, 1 c.c. of rmal alkali = ·019 gramme of fluorine. The process gives good results even in pre- nce of organic matter or volatile acids such as hydrochloric acid, and the author hopes at owing to its simplicity, the process will attract attention; but we fear that not- ithstanding the excellent test analysis, few practical analysts will try it on account of e very complicated apparatus required.

L. de K.

Estimation of Free Lime in the Black Soda Ash. G. Lunge. (*Zeitschr f. ngew Chemie*, October 1st, 1890).—This estimation is of some importance, as the pre- nce of some free lime is indispensable to the complete extraction of the soda. The thor gives his modification of Winkler's process, which, he thinks, will be found useful the assay of black ash. Fifty grams. of the well-mixed sample are quickly powdered a mortar, put into a 500 c.c. flask, and mixed with warm water, which must be free om carbonic acid. It is necessary to frequently shake the flask, otherwise a hard cake ill form at the bottom. After two hours the liquid is made up to the mark, and ter thorough shaking 5 c.c. of the turbid liquid are pipetted off, put into a aker, and mixed with a slight excess of barium chloride. After adding a few drops phenol-phthalein, the alkalinity (lime) is now titrated with $\frac{n}{5}$ oxalic acid. The thor found it totally unnecessary to add sugar, a plan proposed to better dissolve the me.

L. de K.

Assay of Logwood. Dr. L. Schreiner. (*Chem. Zeit.*, No. 58, 1890).—The author d not intend to publish his process, which he has for years successfully employed; but many correspondents believe, the accurate assay of logwood is still an impossibility, he has finally decided to make his process known. Fifty grams. of the ground wood are completely exhausted with water, the liquid diluted up to 1 litre, and a portion of it filtered through hide powder, which must be of sound and woolly quality. The filtrate is generally colourless, and even remains so after addition of ammonia. 100 c.c. of this solution, and also 100 c.c. of the original solution, are evaporated to dryness at 100° C, and the difference between the two weighings represents the amount of colouring matters, including, of course, tannin. For the assay of extract of logwood, 10 c.c. of the powdered sample are dissolved in water and diluted up to 1 litre. To see if there i any adulteration with mineral matter, the ash is taken as well. L. de K.

Testing for Arsenic by Feleitmann's Method.—Professor Johnson has recently shown the great advantage of employing aluminium instead of zinc in this test, because commercial aluminium never contains arsenic, while it is very difficult to obtain zinc that is absolutely free from this impurity. The evolution of hydrogen from aluminium and potassium hydrate also proceeds with great regularity and witho application of heat to any extent.

Detection of Wood-pulp in Paper. Wurster, Maandbl Vervalsch, No. 189.—By means of the following reagents, which must be applied in the presence of an acid, either in the cold or at a gentle heat, the presence of wood-pulp in paper may be readily detected.

Reagent.	Wood-paper.	Cellulose.
Orcine.	Dark red.	No colour.
Resorcine.	Dark green.	Violet.
Pyrogallol.	Greenish blue.	„
Phenol.	Greenish yellow.	„
Phloroglucine.	Blueish violet.	No colour.

Another excellent reagent is dimethyl-paraphenylene-diamine, which colours the wood intensely fuchsia red, and enables the analyst to approximately estimate its quantity.

Popular Chemistry.—The following specimen of chemistry for the million has appeared in the *Echo,* and we reproduce it for the delectation of our readers :—Sixty samples of milk, a dozen samples of flour, bread, meat extracts, potted meats, feeding cakes, etc., can be estimated with the greatest facility, as regards flesh-forming constituents, in an hour. The cost is almost *nil.* One grain of permanganate of potash in solution made acid constitutes sixty workable measures, or *subnormes.* With a slight alteration of the process, alcohol, starch, mucilage can be estimated in the same rapid manner. The cost is so insignificant that a million may be made for a guinea. The only apparatus required are a pipette and flask, and a phial and bottle may be substituted even for these. The calculation required is quite within the capacity of a Board School lad of the sixth standard. The process has lately been brought under the notice of the Agricultural Department and School Board authorities, and will speedily make an impression on vestries, as it has already done in the grain trade. Details may be found in various agricultural papers of the month notably the *North British Agriculturist* of the 22nd and 29th ult., and subsequently. The whole of the experiments may be performed by every householder, and the pipette and flask will take their place the same as scales and weights amongst household requisites. Never has the individual possessed such power over the value of his food stuffs. A kernel of wheat, a few grains of bread, a few drops of milk, such is the test of vegetable and animal albumens.